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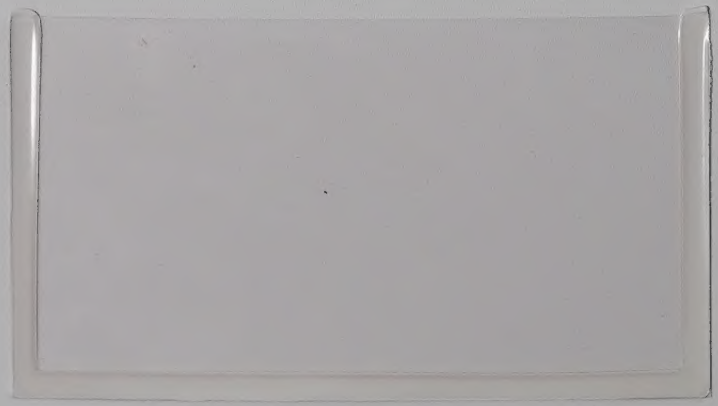
MA32818

DIONEX



DIONEX

# DX-100 ION CHROMATOGRAPH OPERATOR'S MANUAL

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Document No. 034364
Release 4
November 1991

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**PRINTING HISTORY**

Release 1, May 1990
Release 2, October 1990
Release 3, September 1991
Release 4, November 1991

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# SECTION 1
# INTRODUCTION

## 1.1 DX-100 System Overview

The Dionex DX-100 integrated ion chromatography system can perform a multitude of isocratic ion chromatography applications using conductivity detection. You can accomplish both chemically suppressed and nonsuppressed applications with this system.

The DX-100 is designed for ease of use and versatility. The system can be operated manually from the front panel controls, or in an automated mode with the Dionex 4400 Integrator or the Dionex AI-450 Chromatography Automation System. You can also connect an auto sampler to provide total system automation.

## 1.2 Operator's Manual Overview

This manual contains the information you need to install, operate and maintain your DX-100.

**Section 1, Introduction,** gives an overview of the contents of the manual and an introduction to the structure and operation of the DX-100 system.

**Section 2, System Description,** details the operation and function of the DX-100 System components and controls.

**Section 3, Installation,** gives step-by-step system installation instructions. Follow these instructions carefully to ensure ease of installation and proper system operation after installation. This section provides the information necessary to install all components except columns and suppressors.

**Section 4, Operation,** gives column and suppressor installation instructions, and details priming and check-out procedures. This section also provides procedures for sample handling, running analyses, and system shutdown.

**Section 5, Maintenance and Service,** provides routine maintenance information. It also presents a Troubleshooting Guide that lists problems which you might encounter while using the DX-100. Immediately following each problem is a list of possible causes and solutions to the problem. Finally, this section provides service and repair procedures.

The **Appendixes** include additional information of interest to the user: a discussion of conductivity detection principles, ion chromatography terms, 4400 Integrator programming, and a listing of system specifications.

# SECTION 2
# SYSTEM DESCRIPTION

Figure 2-1
Front View DX-100

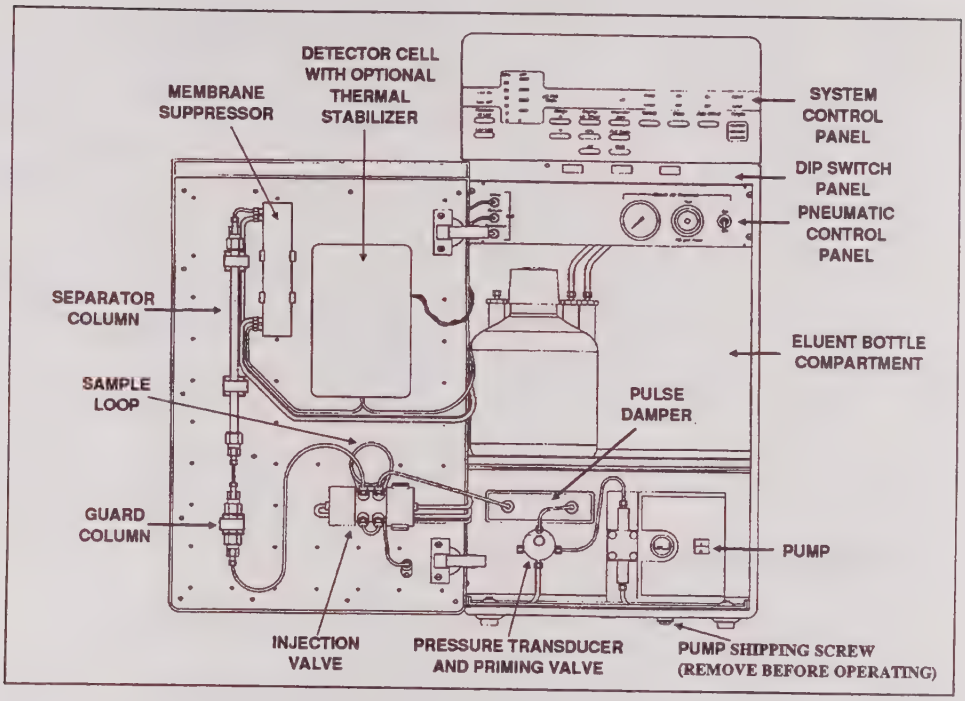
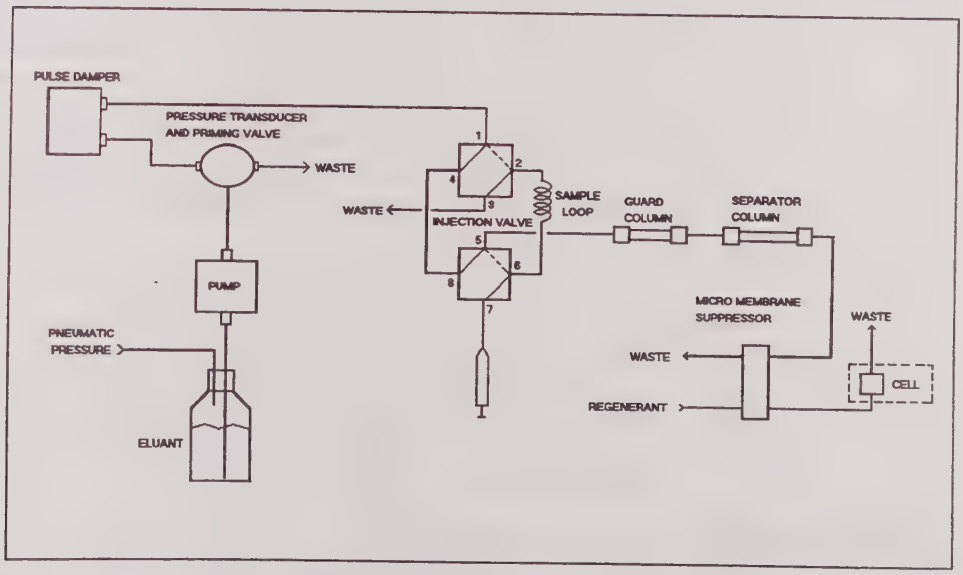
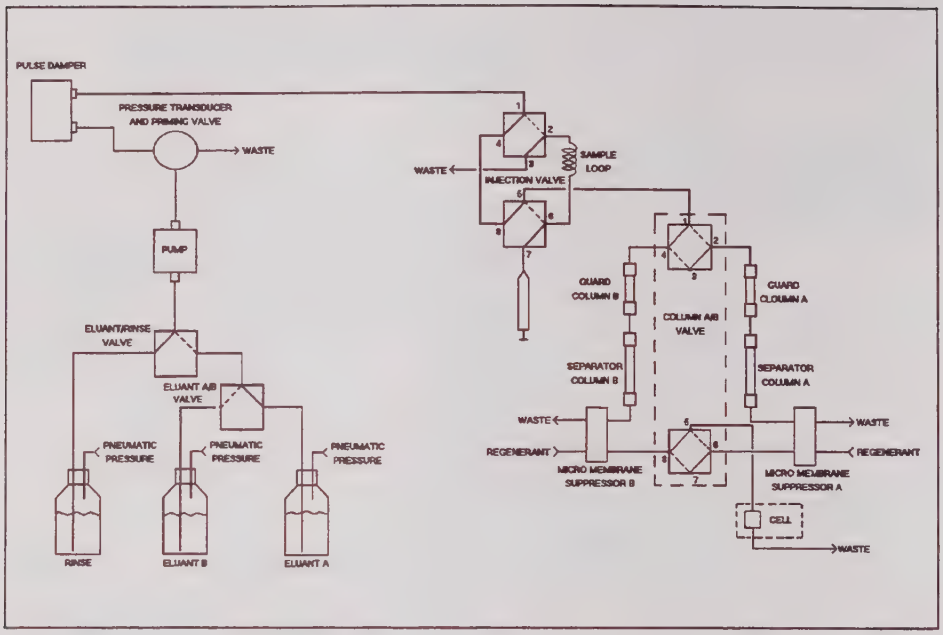



Figure 2-2
DX-100 One Column Set System Flow Diagram




The system shown in Figures 2-1 and 2-2 is configured as a one column set system with one suppressor. Figure 2-3 shows a two column set system with two suppressors. The *basic concept* of operation when processing a sample is the same for both configurations.

Figure 2-3
DX-100 Two-Column
Set System Flow
Diagram




## 2.1 CHROMATOGRAPHY STREAM

### 2.1.1 Eluent Flow

The eluent flows from the eluent bottles, in the upper portion of the system cabinet, to the pump. The eluent is then pumped through the priming valve assembly and pulse damper to the injection valve. A sample is loaded into the injection valve in a fixed volume loop, and is then injected into the chromatography stream. The stream then goes to the guard column and the separator column. When using chemical eluent suppression, the stream flow is from the separator column to the suppressor and then to the detector cell. If you use a non-suppressed application, the chromatography stream goes directly from the separator column to the cell for detection.

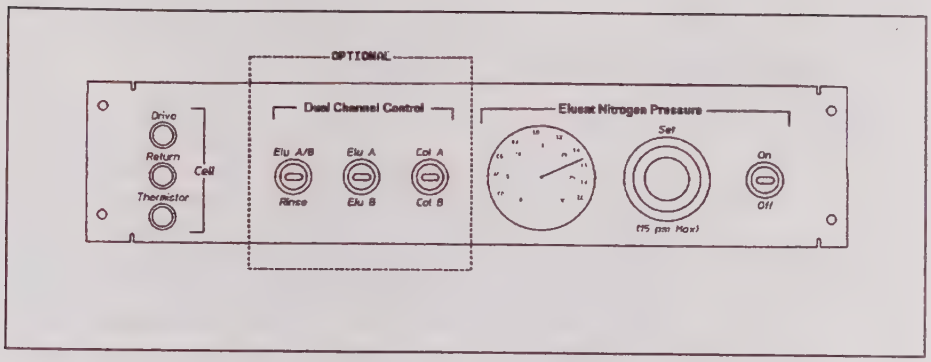
### 2.1.2 Regenerant Flow

Suppressor regenerant enters the system in either of two ways: it can be pumped from an outside AutoRegen unit through the lower rear panel of the DX-100, or it can come from a regulated, pressurized regenerant bottle located outside the system. A 4-liter regenerant bottle can be placed diagonally inside the eluent bottle compartment if desired. Tubing is routed to the regenerant inlet on the suppressor, and from the suppressor outlet to the AutoRegen unit or to waste.

## 2.2 PNEUMATICS CONTROL PANEL

The Standard Pneumatics Control Panel, shown in Figure 2-4, contains a regulator for setting eluent gas pressure and a gauge to monitor that pressure. The two column set Pneumatic Control Panel additionally includes switches which control eluent routing and the column selection valve.

Figure 2-4
Nitrogen Pressure
Control Panel




## 2.2.1 Eluent Nitrogen Pressure

**On / Off Toggle Switch** starts and stops the flow of nitrogen to the eluent bottles.

**Pressure Regulator** controls the nitrogen pressure to the eluent bottles. The maximum setting for this regulator is 15 psi.

**Pressure Gauge** displays the actual nitrogen pressure to the eluent containers.

## 2.2.2 Dual Channel Control

**Elu A/B / Rinse** Toggle Switch allows you to select either eluent or rinse flow.

**Elu A / Elu B** Toggle Switch allows you to select flow from the Eluent A or Eluent B bottle.

**Col A / Col B** Toggle Switch directs eluent flow to either column A or column B.

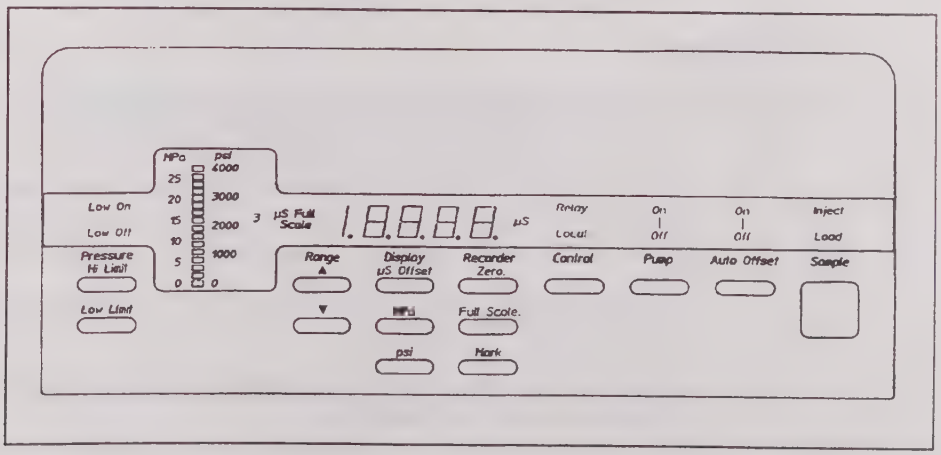
## 2.2.3 Cell Connections

The Cell *Drive*, *Return*, and *Thermistor* connectors, located at the left side of the panel, should require no user interaction other than confirming that they are firmly connected.

# 2.3 SYSTEM CONTROL PANEL

The System Control Panel contains the major switches and displays with which you control and monitor DX-100 operation.

Figure 2-5
DX-100 System
Control Panel



### 2.3.1 Pressure Hi Limit / Low Limit

**Hi Limit**

The Hi Limit button is used both to display and to define the maximum allowable pump pressure. This pressure can be set, in incremental values of 2.7 MPa (400 psi), from 6.9 MPa (1000 psi) to 26.2 MPa (3800 psi). If the selected maximum pressure is exceeded during operation, three things happen: 1. the LED bar gauge above and to the right of the switch flashes on and off at the selected pressure; 2. the pump is turned off; and 3. a TTL high signal is sent to the connector contact labeled Press Alarm. on the DX-100 rear panel. This pressure alarm signal then goes either to a user-supplied alarm or to the 4400 integrator if one is installed.

Briefly depressing this switch causes the user-selected maximum allowable pump pressure to be displayed on the bar graph.

To set the maximum allowable pump pressure, depress the switch until the LED bar increments to 3800 psi. The pressure reading will then start to decrement. When the LED bar reaches the desired limit, release the button. Although the pump operating pressure limit is 4000 psi, the system pressure limit is about 2500 psi; this is approximately 1000 psi higher than typical operating condition pressures. Dionex recommends that the high limit be set at 2500 psi. If an alarm occurs, it must be reset by momentarily depressing the switch.

**Low Limit**

The minimum allowable pump pressure is permanently set at 200 psi. If the pump pressure falls below this value, and the Low Limit switch is On, three things happen: 1. the LED bar gauge flashes off and on at the bottom of the scale; 2. the pump is turned off; and 3. a signal is sent to the connector contact labeled Press. Alarm on the DX-100 rear panel. This signal then goes either to a user-supplied alarm or to a 4400 Integrator if one is installed.

The Low Limit switch mode is indicated either by the words Low On or Low Off displayed above the switch. If an alarm occurs, reset it by turning the switch off and then on.

### 2.3.2 Range

The *Range* buttons control the setting of the detector full-scale range. The range value is expressed in microsiemens (µS). Eleven range values are available: .01, .03, 0.1, 0.3, 1, 3, 10, 30, 100, 300, and 1000 µS. Increase the range by pressing the up-arrow switch; decrease the range by pressing the down-arrow switch.

### 2.3.3 Display

The cell conductivity digital display is located directly above the *Display* button switches. The standard display is cell conductivity in microsiemens.

Pressing the **µS Offset** button replaces the standard display with a read-out, in microsiemens, of the amount of background that is currently being offset (i.e., the offset value when the Auto Offset button was last pressed — see paragraph 2.3.7, Auto Offset).

Pressing the **MPa** button temporarily replaces the standard display with a read-out of cell pressure in Megapascal.

Pressing the **psi** button temporarily replaces the standard display with a read-out of cell pressure in pounds per square inch.

### 2.3.4 Recorder

The three recorder buttons — *Zero, Full Scale,* and *Mark* — are used to send calibration and mark signals to a chart recorder.

Press the **Zero** button to set zero on your chart recorder (0-volt output).

Press the **Full Scale** button to set full scale on your chart recorder. (The output voltage depends upon DIP switch settings which are described in Section 4 of this manual).

Pressing the **Mark** button causes the chart recorder graph line to jump to a point equal to ten percent of the full scale value. Press the *Mark* switch at the same time that you press the *Sample* inject switch, and the resulting mark will indicate the point of sample injection. The functions of the Mark and Sample switches can be combined by setting a DIP switch described in Section 4.1.2.

### 2.3.5 Control

The **Control** button transfers system control between Local and Relay modes. In Local Mode, you control the system from the System Panel. In Relay Mode, some functions are controlled by the 4400 integrator. The current mode of operation is displayed directly above the switch.

### 2.3.6 Pump

Pressing the **Pump** button turns the pump on or off. Either the word *On* or the word *Off* is displayed immediately above the switch.

### 2.3.7 Auto Offset

The **Offset** button allows you to offset background conductivity, thereby zeroing your chromatogram baseline. Press the *Offset* button to toggle offset correction on or off. Set this control when there is no sample in the chromatography stream and the baseline has equilibrated. If you have just turned on the DX-100, allow the system to run for a few minutes before using this control. The value to be offset appears in the main digital display. Press the *Offset* button, and the conductivity display will read zero, indicating that the current background value has been offset. You can display the offset value by pressing the *Display µS Offset* switch; the offset value is displayed immediately above the switch (see paragraph 2.3.3).

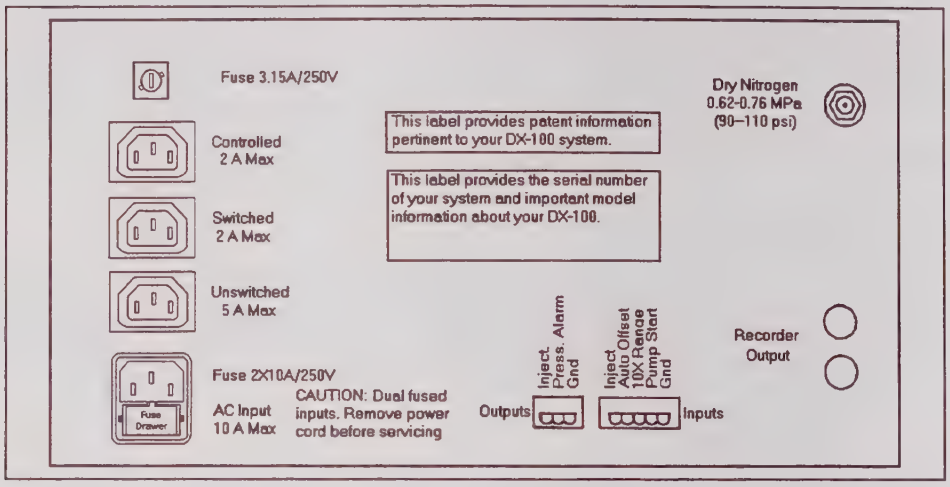
### 2.3.8 Sample

The **Sample** button is used to inject a sample. Pressing this button switches the inject valve between the load and inject positions. A display directly above this switch indicates whether the valve is in *the Inject* or *Load* position.

## 2.4 REAR PANEL

There are three AC outlets at the left of the rear panel. The bottom outlet is active as long as the DX-100 is plugged into an AC outlet; the middle outlet is active only when the DX-100 power switch is on; and the top outlet is active only when the System Control Panel pump switch is on.

**Figure 2-6**
**Rear Panel**



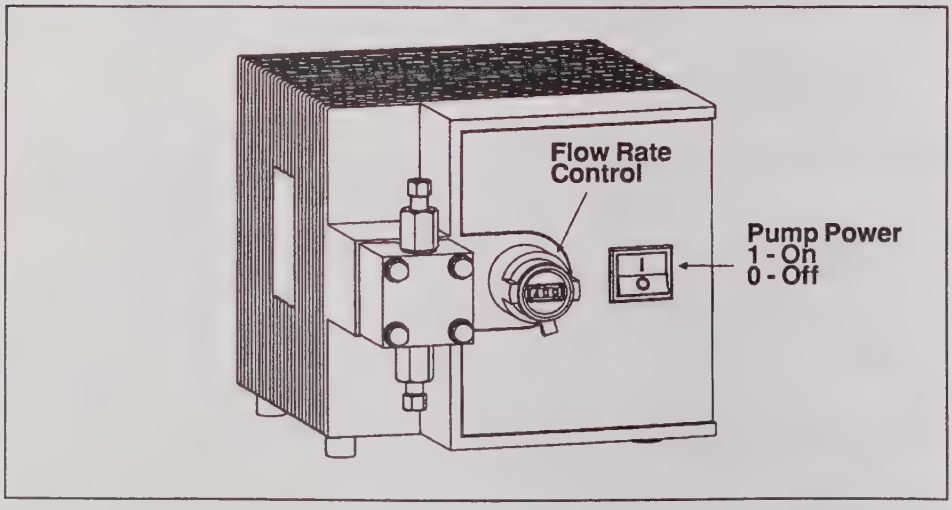

The other connections on this panel are described in detail in Section 3, Installation.

## 2.5 PUMP

The location of the pump in the DX-100 is shown in Figure 2-1. The pump is illustrated in Figure 2-7

The single piston pump delivers a flow rate from 0.5 to 4 ml/min, adjustable in increments of 0.1 ml/min by the control knob shown in Figure 2-7. Setting pump flow rate is discussed in Section 4, System Operation.

**Figure 2-7**
**DX-100 Pump**



**NOTE: During shipment the pump is held in place by a shipping screw (see Figure 2-1, page 2-1 for location). To prevent vibration, this screw must be removed prior to operating the pump. Before shipping or transporting the DX-100, reinstall the screw to prevent damage to the rubber isolation feet.**

## 2.6 CONDUCTIVITY CELL

The flow-through cell has an active volume of 1.25 µL. The cell body is a high-density polymeric material containing two passivated 316 stainless-steel electrodes. The electrodes are permanently sealed into the cell. The detector cell constant is calibrated electronically. A thermistor located slightly downstream from the electrodes measures the temperature of the liquid inside the cell; this measurement is then used for temperature compensation.

# SECTION 3
# INSTALLATION

## 3.1 UNPACKING AND INSPECTION

Inspect the DX-100 for shipping damage as soon as you receive it. Many carriers limit their claim period to 10 days after delivery, so report any shipping damage to the incoming carrier immediately. To get help in assessing shipping damage or to report a missing or damaged part, contact your nearest Dionex office.

The shipping kit contains all parts needed to install and maintain the SCM. The contents of the shipping kit are listed on the packing list. Check the contents of each package against the packing list to verify that you have received all components.

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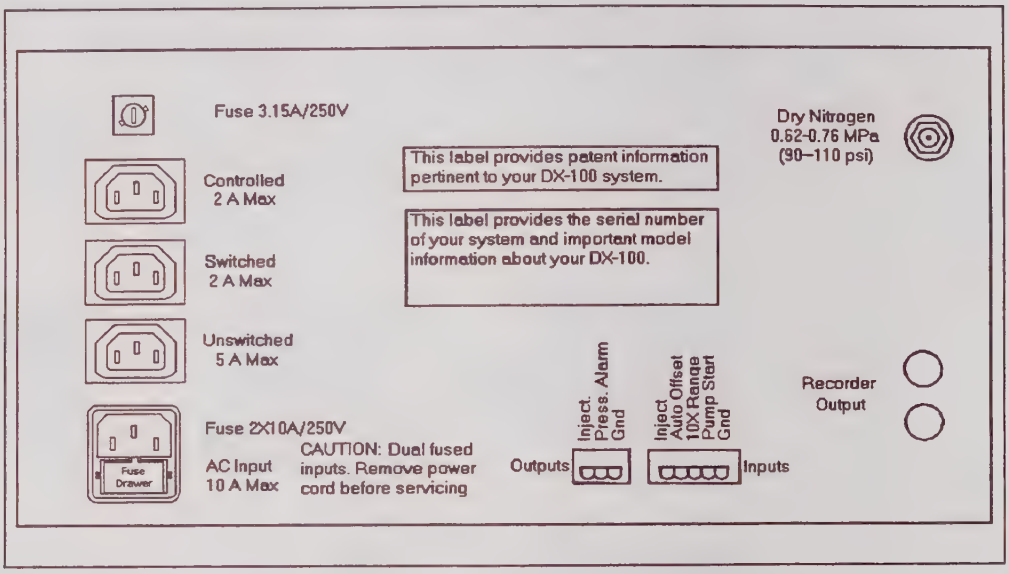
FAX 81-6-885-1215

**NOTE: Before starting the installation, remove the pump shipping screw described in Section 2.5 of this manual.**

## 3.2 FACILITIES REQUIRED

Figure 3-1
Rear Panel
Connections




### 3.2.1 Bench Space

Place the DX-100 on a sturdy laboratory bench at least 60 cm (23 1/2 inches) deep. Additional bench space is needed if you use a recorder or an integrator.

### 3.2.2 Power

The DX-100, depending upon model, requires a grounded 100 VAC. 115 VAC, 230 VAC, or 240 VAC single-phase power source. The line frequency is factory-set to either 50 or 60 Hz. The three-conductor line cord ensures a safety ground. Check the power configuration of your system on the system data label on the back panel to confirm that the system is compatible with your laboratory. If you are unsure that it is compatible, consult a professional electrician.

### 3.2.3 Pneumatic Pressure

The sample injection valve is pneumatically operated. Use a clean laboratory nitrogen source or a nitrogen cylinder. Install an appropriate regulator on the nitrogen supply to adjust the pressure to the required 62MPa (90psi) to 76 MPa (110 psi).

### 3.2.4 Deionized Water

Use only high quality deionized water for the preparation of aqueous eluents and for rinsing procedures. The water must be ASTM filtered Type II (1 megohm or 1 μS) or better. The use of high quality water reduces background interference. This reduction is desirable for most analyses and essential when performing trace-level analyses. All Dionex specifications are based on ASTM Type I water having a background resistivity of at least 16.67 megohm (0.06 mS or less conductivity).

To avoid contamination and plugging of the columns and flow system, the water must contain no particles larger than 0.2 microns. Several deionization/filtration systems are commercially available for purchase or lease. Make sure an appropriate water supply is available before beginning operation of the DX-100. For additional information, contact your Dionex Customer Satisfaction representative (Section 3.1).

## 3.3 ELUENT BOTTLE CONNECTIONS

### 3.3.1 Pneumatic Tubing

Untape the coil of red pneumatic tubing hanging in the eluent bottle compartment. Slip the end of the tube firmly down over the barbed Qik Connect fitting on the eluent bottle. If you have a two column set system, three pneumatic tubes are provided, one for each bottle.

### 3.3.2 Eluent Tubing

Untape the coil of white eluent tubing hanging in the eluent bottle compartment. Screw the tube end-fitting securely into the eluent tube fitting on the bottle. If you have a two column set system, three labeled eluent tubes are provided, one for each bottle.

## 3.4 REAR PANEL CONNECTIONS

### 3.4.1 External Pneumatic Tubing Installation

1. Locate the white barbed gas fitting (P/N 030077) and the pipe adaptor (P/N 030087) in the Shipping Kit. Screw the barbed fitting into the outlet of your nitrogen supply regulator. Or, if the regulator outlet is incompatible with the 10-32 thread on the barbed fitting, first screw the pipe adaptor into the nitrogen supply regulator and then screw the barbed fitting into the pipe adaptor.

2. Locate the coil of red 1/8 inch OD gas tubing (P/N 030092) in the Shipping Kit. Push one end of the tubing over the barbed *Dry Nitrogen* fitting on the rear panel of the DX-100. Push the other end over the barbed fitting attached to the nitrogen supply regulator.

3. Open the door of the DX-100 to gain access to the Nitrogen Control Panel. Turn the *Eluent Nitrogen Pressure* switch *On*. Turn on the cylinder or laboratory nitrogen supply and adjust the regulator to between 0.69 MPa (90 psi) and 76 MPa (110psi).

4. Next, press the Sample button on the System Control Panel and listen for escaping gas. An initial burst of gas is released each time the Sample switch is pressed; this sound is normal. A continuous sound of escaping gas indicates a leak. If necessary, press the Sample switch several times until no sound of escaping gas is heard. If a gas leak persists, refer to Section 5.3.5.

### 3.4.2 Waste Lines

Locate the waste lines exiting through the Fluid Line Slot in the rear panel. Place the free ends of these lines into an appropriate waste container. To avoid waste siphoning back into the system, the ends of the waste lines must be below the bottom of the analyzer.

### 3.4.3 Regenerant Lines

If you use an external Autoregen accessory or regenerant bottle (from the MMS installation kit) connect the input and return fluid lines from the regenerant source to the labeled input and output lines extending from the rear panel of the DX-100.

### 3.4.4 Electrical Connections

#### 3.4.4.1 AC Power Outlets and Cables

There are three AC power outlets at the left side of the rear panel.

**The bottom outlet**, marked *Unswitched,* is active as long as the DX-100 is plugged into an external AC outlet.

**The middle outlet,** marked *Switched,* is active only when the DX-100 Power switch is on.

**The top outlet**, marked *Controlled,* is active only when the System Control Panel Pump switch is *On.*

The DX-100 is is supplied with two cables for use with these IEC 320-2-2E compatible connectors: an accessory jumper and an accessory power adapter.

The **accessory jumper cable** can be used to provide AC power to other modules which use IEC 320 connectors. (To order additional accessory jumper cables specify Cable, Linecord, Jumper, P/N 960748)

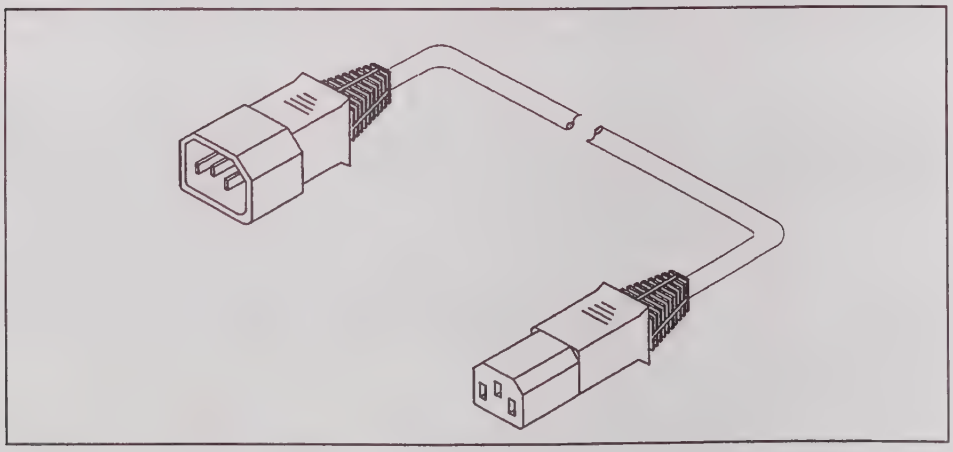
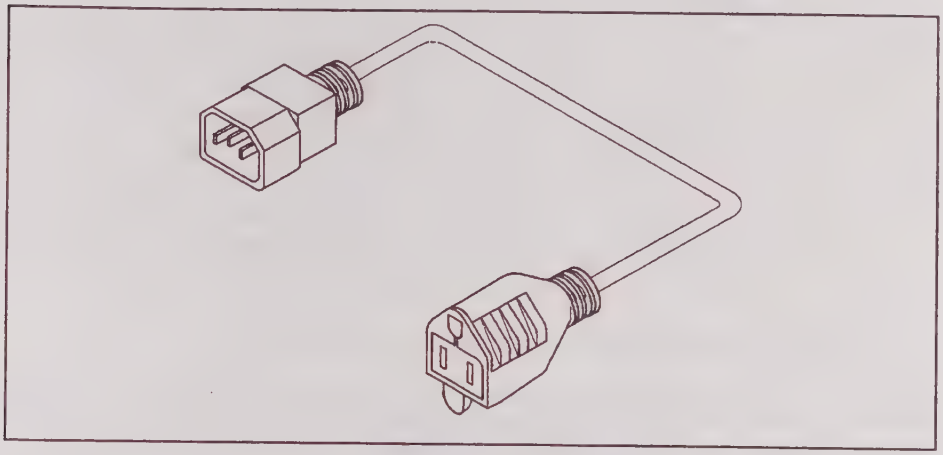


**Figure 3-2 Accessory Jumper Cable (IEC 320-2-2E Compatible)**

The **accessory power adapter cable** is used to power modules which use NEMA 5-15 connectors (the common 3-prong American line cord plug). (To order additional accessory jumper cables specify Cable, Linecord, IEC/NEMA 5-15R, P/N960749)

**Figure 3-3 Accessory Power Adapter Cable (IEC/NEMA 5-15R)**



IEC 320 adapters compatible with the connector standards of countries which do not use the connectors noted above are generally available from local electrical hardware suppliers. These suppliers can also provide rewireable IEC 320 accessory plugs which can be installed on existing cables by an electrician.

### 3.4.4.2 Power Supply

1. Make sure the system *Power* switch located on the upper-right side of the DX-100 is in the 0 (zero/off) position.

2. On the upper rear panel of the DX-100, locate the label which notes the line frequency (50 or 60 Hz) and Voltage (100, 115, 230, or 240 VAC) for which the unit was designed. **Make sure that the frequency and voltage noted on the label are appropriate for your location. (See paragraph 3.2.2)**

3. Remove the AC Power Cable (P/N 960708) from the shipping kit. Plug the cable"s female connector into the rear panel connector labeled *AC Line*. Plug the cable's male connector into your external power source. Turn on the *Power* switch noted in Step 1, above.

### 3.4.4.3 Line Input Fuse

Two fuses in the power entry connection limit to 10A the total power drawn by the DX-100 and auxiliary loads. A failed fuse indicates that a fault has occurred in the DX-100 or in an auxiliary unit connected to a DX-100 rear-panel AC outlet. **NOTE:** If a new fuse fails immediately, or soon after replacement, your DX-100 probably needs professional servicing. Contact your nearest Dionex Customer Satisfaction Representative for assistance (paragraph 3.1).

Line input fuses are located in the fuse drawer, located immediately below the socket on the AC input connector (see Figure 3-1). To replace fuses, first remove the AC line cord, and then remove the fuse drawer by squeezing the retaining clips located at the right and left of the drawer and pulling the drawer outward. Replace both fuses. Spare

fuses are furnished in the shipping kit. The fuses are 5 X 20 mm, 10A/250V, high interrupt (breaking ) capacity type (IEC 127, sheet1).

#### 3.4.4.4 Controlled AC Fuse

The controlled AC outlet and the internal eluent pump share a common controlled AC branch circuit. To protect the controller, a 3.15 amp fuse is present to interrupt the live pole of this branch. To replace the fuse, use a small flat-bladed screwdriver or a small coin to turn and release the bayonet fuse-holder. A spare fuse is furnished in the shipping kit. The fuse is a 5 X 20 mm, 3.15 A/250V high interrupt (breaking) capacity type (IEC 127, sheet 1). These fuses are opaque and can be checked with an ohmmeter or other continuity-checking device. **NOTE:** If the new fuse burns out immediately, or soon after replacement, your DX-100 probably needs professional servicing. Contact your nearest Dionex Customer Satisfaction Representative for assistance (paragraph 3.1).

### 3.4.5 Chart Recorder Connections

To connect a chart recorder, first connect one end of the Analog Signal Wire Assembly (P/N 043598) to the Recorder Output connectors on the DX-100 back panel (see Figure 3-1). Then connect the other end of the cable to the appropriate input connectors on your chart recorder.

### 3.4.6 4400 Integrator Connections

#### 3.4.6.1 Analog Data Transmission

In its simplest operating mode, the integrator can be used as a chart recorder with integration capabilities. If this mode is chosen, the 4400 Integrator is connected using the Analog Signal Wire Assembly (P/N 043598) found in the DX-100 Ship Kit. Connect one end of the cable to the Recorder Output connectors on the DX-100 back panel, and the other end of the wires to 4400 Integrator Input Connectors.

#### 3.4.6.2 Time Functions Automation

You can greatly enhance the automation capabilities of your system by purchasing and using a DX-100 Automation Cable. For installations consisting of one DX-100 and one autosampler used with one integrator, you must use a special cable (Automation Cable, DX-100 to Integrator to ASM - P/N 043051). For installations consisting of two DX-100s and two autosamplers used with one integrator, you must use a different cable (Automation Cable, 2 DX-100 to Integrator to 2 ASM - P/N 043069). Before installing the automation cable between the DX-100 and the integrator, confirm that the front panel *Automation* button is switched to the *Local* position. The button should be switched to the *Relay* position only after the integrator *Time Functions* have been set to initial conditions (Appendix C). To connect the DX-100 Automation Cable, first connect the cable's 36-pin connector to the 4400 Integrator *Time Functions* output connector. Then connect the Automation Cable's 3-pin male green connector to the green *Outputs* connection on the DX-100 back panel (Figure 3-1). Next, connect the Automation Cable's 5-pin male green connector to the *Inputs* connection on the DX-100 back panel (Figure 3.1). If a wire becomes detached from a green plug, reconnect it by loosening the small screw at the top of the wire connection port on the green connector, inserting the wire in the port, and retightening the screw. Connections are detailed in the following chart.

*Outputs*

| | | |
|---|---|---|
| Inject | Start Run Channel A | Orange |
| Press. Alarm | Bit 3 BCD Data Input | Blue |
| Ground | Ground | Black |

*Inputs*

| | | | |
|---|---|---|---|
| Inject | T6 | Channel A | Yellow |
| Auto Offset | T5 | Channel A | Brown |
| 10X Range | T4 | Channel A | Gray |
| Pump Start | T7 | Channel A | Green |
| Ground | Ground | | Red |

### 3.4.6.3 Automated Sampler Module (ASM)

To install an ASM, first attach the 50-pin parallel connector from the DX-100 Automation Cable (P/N 043051 or 043069 - see Section 3.4.6.2) to the ASM back panel port labeled *Parallel Interface*. Next, plug the telephone-type connector from the Cable into the ASM back panel port labeled *Relay*. For further information see the manual that was supplied with your ASM.

## 3.4.7 AI-450 Chromatography Workstation Connections

The DX-100 can be used with the AI-450 Chromatography Workstation to provide automated system control, data acquisition, data processing, and report generation.

If samples are loaded manually with a syringe, each analysis run is started manually from the DX-100, the Advanced Computer Interface (ACI), or the AI-450 computer program.

If an automation cable (Automation Cable, 4 function, DX-100 to ACI - P/N 043070) is installed between the DX-100 and the ACI, the AI-450 can control time functions (injection, offset, range change, pump on/off) for each of the analysis runs once they are started. If the Automation Cable is not installed, the runs can only be started with the *Sample* injection button, and the AI-450 acts only as a data collection and reporting device.

If samples are loaded using an autosampler, the Automation Cable (P/N 043070) is used between the DX-100 and the ACI, and the AI-450 automatically performs the whole schedule of analyses. The schedule, which is programmed into the AI-450, is started from the computer or ACI once the samples are loaded into the autosampler.

### 3.4.7.1 Analog Data Transmission

When using an AI-450, DX-100 detector data is transmitted to an ACI, which converts the analog data to digital and sends it to your computer for storage. A Coaxial Cable Assembly and a special connector must be installed between the DX-100 and the ACI to transmit this data.

**Install the Analog Data Coaxial Cable Assembly as follows:**

1. Locate one of the two male-to-male coaxial cables (P/N 013274) in the ACI Ship Kit. Then locate the female coax/double banana plug (P/N 037332) in the DX-100 Ship Kit.

| DX-100 CONNECTION | INTEGRATOR FUNCTION | WIRE COLOR |
|---|---|---|

2. Connect the end of the coaxial cable to the coaxial connector on the coax/double banana plug. Insert the double banana plug into the *Recorder Out* connectors at the right side of the DX-100 back panel. **Make sure that the banana plug jack on the side of the plug labeled GND is inserted into the black *Recorder Out* connector.**

3. Connect the unattached end of the coaxial cable to the desired *ANALOG INPUT* on the back panel of the ACI.

### 3.4.7.2 Manual Sample Loading without the Automation Cable

If you load samples manually with a syringe and do not use an automation cable, a signal wire must be installed between the DX-100 and the ACI to transmit a start signal when the *Sample* button is switched to the *Inject* position. The wire, an Analog Signal Wire Assembly (P/N 043598), is provided with your DX-100. This wire is not used for detector data with the AI-450; the cable described in Section 3.4.8.1 is used instead.

**Install the Signal Wire Assembly as follows:**

1. Locate the Analog Signal Wire Assembly (P/N 043598) in the DX-100 Ship Kit.

2. Connect a red insulated wire to the *Inject* contact of the *Output* connector on the DX-100 back panel. Connect the adjacent black insulated wire to the *Gnd* contact of the Output connector on the DX-100 back panel.

3. Fasten the unattached end of the red insulated wire to either the *RUN SYS1* connection (the first pin from the top of the 8-pin green connector) or the *RUN SYS2* connection (the fifth pin from the top of the 8-pin green connector) on the ACI back panel. Fasten the unattached end of the black insulated wire to the *GND* connector on the ACI back panel.

### 3.4.7.3 Fully Automated Analysis Using an Autosampler

Complete system control and unattended analysis capability requires a special cable (Automation Cable, 4 Function, DX-100 to ACI, P/N 043070) connecting the DX-100 and the ACI. To connect the cable, first attach the cable's 5-pin male green connector to the *Inputs* connection on the DX-100 back panel (Figure 3-1). Next, attach the cable's 16-pin male green connector to the 16-pin RLY/TTL connection on the ACI back panel. The DX-100 is fully capable of receiving relay (RLY) or transistor transistor logic (TTL). Connections are detailed in the following chart. Before connecting the autosampler to the ACI refer to the ASM and ACI User's Guides. To program the AI-450, refer to the AI-450 User's Guide.

| DX-100 CONNECTION | ACI CONNECTION |
|---|---|
| Inject | RLY 1 or TTL 1 |
| Auto Offset | RLY 2 or TTL 2 |
| 10X Range | RLY 3 or TTL 3 |
| Pump Start | RLY 4 or TTL 4 |
| Ground | Ground pins for all relays |

## 3.5 PROGRAMMING THE 4400 INTEGRATOR

If you have a 4400 Integrator and a Dionex Automated Sampler, you can completely automate your analyses by entering a short BASIC language program into your integrator. This program gives the integrator the capability to fully monitor the status of the automated sampler and DX-100, and to coordinate injections. The software program directs the integrator to monitor such conditions as the readiness of the injection valve to load and the status of the automated sampler — Is it in the middle of a rinse procedure? Is it loading another sample? Is it ready for an inject command? The software program can also direct appropriate action if a pressure alarm occurs.

Appendix C provides detailed information and loading procedures for the 4400 Integrator software program.

# SECTION 4
# OPERATION

## 4.1 APPLICATION INSTALLATION

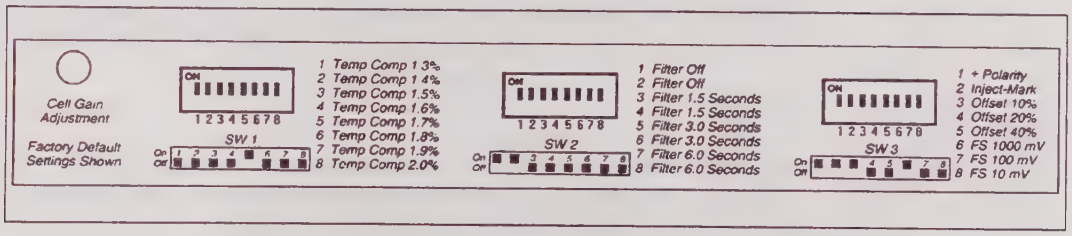
This section provides the procedures used to install a new application in the system, and details the steps required to start up the system after it has been shut down for several days.

### 4.1.1 Reagent preparation

The eluents and regenerants used are determined by the application installed. Refer to the column and suppressor manuals and to the Application Note information on preparation. Transfer the prepared eluent to the appropriate 2 L eluent bottle in the DX-100. Transfer the regenerant to the 4 L bottle of the Micro Membrane Suppressor installation kit, unless an Auto Regenerant Accessory is used.

### 4.1.2 DIP Switch Setting

**Figure 4-1 Dip Switch Panel**




The DIP switches provide control for temperature compensation, noise filter, and recorder settings. The factory default settings are appropriate for the majority of suppressed conductivity applications. You may never change some settings, but others (such as polarity, temp comp, and filters) you may want to reset as you install nonsuppressed applications. Become familiar with the functions of each of the following switches.

**Temp Comp (Switch 1, positions 1 through 8)** - These switches adjust the value of the temperature coefficient used by the detector. The factory default setting of 1.7% (SW1, position 5, up) is appropriate for most eluent solutions. Refer to Section 4.1.7 for information on how to optimize this setting. If more than one *Temp Comp* switch is on, the compensation value is the average of the lowest and highest settings. If no switch is on, the cell drive is disabled and the DX-100 will not function correctly.

**Filter (Switch 2, positions 1 through 8)** - This filter is a 5th order low pass type providing extremely sharp cut-off of higher frequency noise sources. These filters typically are only needed for nonsuppressed applications which have higher backgrounds and noise. One **pair** of the switch positions must be in the up position and all other switches in the down position for the system to work properly. For example, **both** switch positions 3 and 4 must be in the up position for a 1.5-second filter; **both** switch positions 5 and 6 must be up for a 3.0-second filter. The default is Off. (positions 1 and 2 up; positions 3 through 8 down).

5. Ferrule fittings can be used with both PEEK and Tefzel tubing.

6. ThermoFlare fittings cannot be installed on PEEK tubing.

Refer to Section 5.3.1 for instructions on how to install ferrule fittings on PEEK and Tefzel tubing.

A special flaring tool is required to make ThermoFlare fittings. ThermoFlare tools with instructions can be ordered from Dionex. If you prefer not to buy the flaring tool, tubing with factory-made ThermoFlares is available in a range of IDs and lengths (see Section 5.3.1.2).

**Installation**

1. Turn off the pump.

2. Remove and save the plugs from the analytical columns.

3. Remove and save the unions connecting the lines between the injection valve and the detector cell.

4. If you have a one column system, connect the line from the injection valve to the inlet of the guard column. If you have a two column set system, connect the line from the column switching valve to the inlets of the guard columns.

5. Connect the outlet of the guard column to the inlet of the analytical column, using the short length of tubing.

6. For suppressed applications, connect the outlet of the analytical column to the inlet of the suppressor. For nonsuppressed applications on one column systems, connect the outlet of the analytical column directly to the cell. For non-suppressed applications on two column set systems, connect the outlet of the analytical column to the column switching valve.

### 4.1.6 Application Switching on a Two Column System

To change from Column Set A to Column Set B, follow steps 1 through 14, below (Figure 2-3):

1. Turn off the pump and install Set B columns and suppressor eluents (Section 4.1.5).

2. Ensure that the eluent bottles are filled with the appropriate solutions, and that the rinse bottle is filled with deionized water.

3. Pressurize the bottles to between 5 and 10 psi, inclusive.

4. Set the *Elu A/B / Rinse* toggle switch to *Rinse*.

5. Open the bleed screw on the pressure transducer two full turns.

6. Turn on the pump.

7. After the pump has run for ten seconds, close the bleed screw and let the pump continue to run for an additional two minutes.

8. Set the *Col A / Col B* toggle switch to *Col B*.

9. Set the pump flow rate control knob to the correct setting for the application to be run on Column Set B.

10. Set the *Elu A / Elu B* toggle switch to *Elu B*.

11. Set the *Elu A/B / Rinse* toggle switch to *Elu A/B*.

12. Open the bleed screw on the pressure transducer two full turns.

13. Wait ten seconds after opening the bleed screw (step 12). Close the bleed screw and let the system equilibrate.

14. Proceed to to Section 4.2.2, System Startup and Test.

### 4.1.7 Optimizing Temperature Compensation

The temperature compensation setting for most eluents is approximately 1.7%/° C. To minimize temperature-caused variations in conductivity, optimize the temperature compensation setting for each eluent. This is especially important when operating at high sensitivities. The optimum setting for any eluent is determined as follows:

1. Install a 30 cm (12 in) length of 0.5 mm ID tubing (P/N 035519) between the outlet of the suppressor and the cell inlet for suppressed methods, or between the outlet of the column and the cell inlet for nonsuppressed methods.

2. Prepare a beaker of room-temperature water and one of warm (60° C) water. Submerge the tubing from Step 1 in the room-temperature water.

3. Install the columns.

4. Turn on the Temp Comp 1.7% Dip Switch (Figure 4-1).

5. Pump eluent through the system.

6. Select an *Output Range* which keeps the baseline on scale.

7. After the system reaches equilibrium and the baseline stabilizes, set the Output Range to 10 mS. Set Auto Offset. Position the recorder pen about half-way up the paper.

8. Record the baseline for 1-2 minutes.

9. Remove the tubing from the room-temperature water and place it in the warm water for 1-2 minutes. Note the recorder baseline shift. If the baseline shifts positive, the temperature compensation setting is too low. If it shifts negative, the setting is too high.

10. Place the tubing in the room-temperature water and wait 1-2 minutes to allow the baseline to restabilize.

11. Based on the results of Step 9, increase or decrease the temperature compensation setting by 0.1%.

12. Repeat Steps 9 and 10 until the recorder baseline shift is at a minimum when the temperature changes. NOTE: The temperature compensation value is characteristic of the eluent and can be affected by changes in eluent composition.

13. Disconnect the tubing and reconnect the suppressor or column to the cell inlet as it was originally.

## 4.2 SAMPLE ANALYSIS

### 4.2.1 Sample Preparation

Below are some general considerations for handling samples prior to analysis.

**Collection and Storage**

Collect samples in high density polyethylene containers that have been thoroughly cleaned with deionized water. Do not clean containers with strong acids or detergents because they leave traces of ions on the container walls; these ions may interfere with analysis.

Bacteria in the samples may cause the concentrations of ions to change with time, so samples which will not be analyzed the same day they are collected should be filtered through clean 0.45 µm filters immediately after collection. Refrigerating the samples at 4° C minimizes, but does not eliminate, bacterial growth.

Analyze samples containing nitrite or sulfite as soon as possible. Nitrite oxidizes to nitrate, and sulfite to sulfate, thus increasing the measured concentrations of these ions in the sample. Most samples that do not contain nitrite or sulfite may be stored for at least one week in a refrigerator without significant changes in anion concentrations.

**Pretreatment**

Analyze rain water, drinking water, and air particulate leach solutions directly with no sample preparation (other than possibly dilution). Filter groundwater and wastewater samples through 0.45 mm filters before injection if they were not filtered after collection.

If samples may contain high concentrations of organic contaminants such as groundwater and wastewater, filter them through Dionex OnGuard P syringe filters (P/N 039597) before injection. Be sure to activate the cartridge before use by pushing 5 mL of deionized water through the filter.

**Dilution**

The concentrations of analytes in ion samples can vary widely from sample to sample, so no one dilution can be recommended for all samples of one type. However, some samples can be analyzed with no dilution. The analyte concentrations in many water samples are low enough so that dilution is usually not necessary.

The solution used to dilute the sample (the diluant) may be deionized water, but eluent is preferable since diluting an eluent minimizes the effect of the water dip at the beginning of the chromatogram. If eluent is used as the diluant, then eluent should also be used as diluant to prepare the calibration standard. This is most important for fluoride and chloride, anions which elute near the water dip. The accuracy of fluoride determinations at concentrations of less than 0.5 ppm may be improved by diluting standards in eluent or by spiking the samples with concentrated

eluent to minimize the water dip. For example, a 100 mL sample could be spiked with 1.0 mL of a 100 X eluent concentrate (180 mM $Na_2CO_3$, 170 mM $NaHCO_3$).

**Air Particulate Extracts**

Extract air filters in either deionized water or in eluent. Extracting in eluents has the advantage of eliminating the water dip, if there is one, at the beginning of the chromatogram. Use the following procedure for extracting ions from air filters.

1. Place the filter in a container with 25 mL of deionized water or eluent.
2. Place the container in an ultrasonic cleaner and sonicate for 45 minutes.
3. Next, place the filter in a Buchner funnel and wash with three 5-mL portions of deionized water or eluent.
4. Combine the solution used in the sonicator and the washes from the Buchner funnel in a 50 mL volumetric flask and dilute to the mark with deionized water or eluent.
5. Analyze the air particulate extract using the procedures listed above.

## 4.2.2 System Startup and Test

With all the chromatographic and automation hardware installed, and the eluents, standards, and samples prepared, it is time to start up and test the system.

1. Turn the system power on and set the *Control* button on the System Panel to Local.
2. Turn on the system nitrogen and confirm that the pressure is between 62 MPa (90 psi) and 76 MPa (110 psi).
3. Confirm that the eluent and regenerant containers are full.
4. Pressurize the eluent and regenerant containers. If you are using an AutoRegen accessory, turn it on.
5. Check to make sure that the integrator is on and configured to at least print out an area percent report (Method 0).
6. Ensure that the pump flow rate adjustment is correct, and turn on the pump.
7. Set the detector range to the appropriate operating range (typically 30 µS).
8. Enable Auto Offset by pressing the *Auto Offset* button. Allow several minutes for equlibration, then re-press the Auto Offset button to disable Auto Offset. Re-enable Auto Offset by again pressing the Auto Offset button and check that the mS Offset approximates the background expected.
9. If you are using an automated sampler, place it in HOLD. Fill an automated sampler vial with a standard and place it in the automated sampler. Set the automated sampler to RUN.
10. If you are injecting manually with a syringe, fill the syringe with a standard and load the sample loop.

11. Press *Sample* to inject. If you have not installed a DX-100 Automation Cable, you must also press INSERT on the integrator as you inject the sample.

After the run ends, compare the resulting chromatogram with the test chromatogram enclosed with the column or shown in the application literature. Do the chromatograms look essentially the same? If so, go on to Section 4.2.3, Running the Analysis. If the two chromatograms do not look the same, check the flow rates and make a few more injections. The system may need to equilibrate. If you continue to experience difficulties, recheck all system connections. If problems persist, check Trouble-Shooting, Section 5.2, for the symptoms you are experiencing and their remedy. If the problem continues, contact your Dionex Customer Satisfaction Representative (See Section 3.1).

### 4.2.3 Running the Analysis

With the system now running and tested, you are ready to run your calibration standards and samples.

The procedure is similar to that which you just used to test the system. When doing the analysis, run a standard or set of standards first to calibrate the system, then run the samples.

If you are using a 4400 Integrator, calibration standards and samples are run using a DIALOG program with the integrator (see Appendix C). After the system is running and the test injections process successfully, the program is given a command to prepare for calibration. Standards and samples are loaded and the program is started with a sample inject command.

When you are ready to run the standard or standards, type in CA=1 on the integrator. Only type this once for each set of standards.

# SECTION 5
# MAINTENANCE AND SERVICE

## 5.1 ROUTINE MAINTENANCE

It is very important that you perform the following maintenance procedures to keep your DX-100 operating reliably.

1. At least once daily check the interior of the pump compartment for leaks or spills. Isolate and repair leaks, and clean up any spills. Rinse any dried eluent or reagent off components with deionized water.

2. At least once a week check all air lines for crimping or discoloration. Relocate any pinched lines. Replace damaged lines. Empty the waste container to prevent liquid overflows.

3. At least once a week check the junction between the pump head and the metal pump casting for evidence of liquid leaks. Normal friction and wear gradually result in small liquid leaks around the piston seal. If unchecked these leaks can gradually contaminate the piston housing, causing the pump to operate poorly. If leaks are detected, replace the piston seals (Section 5.3.13).

## 5.2 TROUBLE-SHOOTING GUIDE

### 5.2.1 Liquid Leaks

| Possible Cause | Possible Remedy |
|---|---|
| A. Leaking fitting. | Make sure all liquid line connections are tight:<br>1. If the connections are made with Tefzel tubing and ThermoFlare fittings see Section 5.3.1.<br>2. If the connections are made with Dionex ferrule fittings, see the fittings manual (Doc. No. 034213) for instructions<br>3. If you have a two column system, remove the switching valve from its door mounting clip to improve access to all valve ports. Use a wrench on the valve pressure bolt to prevent it from turning while tightening the fitting. |
| B. Broken liquid line. | Cut the tubing at the break and replace the ferrule. Remake the flare (Section 5.3.1), or replace the tubing. |
| C. Blocked or improperly installed waste line. | Make sure the waste lines from the valves and cells are not crimped or otherwise blocked. Also, make sure that the lines are not elevated at any point after they exit the DX-100. |

| | |
|---|---|
| D. Pressure bolt(s) on column injection valve are too loose. | Follow the procedure below to tighten the pressure bolts:<br>1. Turn off the liquid flow.<br>2. Tighten each pressure bolt finger-tight, then use an open end wrench to tighten an additional 1/8 turn. **NOTE:** For optimum leak-free operation, all eight pressure bolts must be evenly tightened against the slider.<br>3. Turn on the liquid flow and check for leaks. Tighten pressure bolts further if leaks appear. **NOTE:** For optimum leak-free operation, use a wrench to evenly tighten pressure bolts. If leakage is still apparent, continue to tighten the bolts in accordance with the values given in the valve manual (Doc. 032678) from the Ship Kit. |
| E. A valve port face is scratched. | Replace the port face (Section 5.3.7). |
| F. Pump check valve housing is loose. | Make sure that the check valves are firmly seated in the pump head. If they are not, tighten them carefully with an open end wrench just until the leak stops (25 in-lb). |
| G. Pump piston seal is damaged. A damaged seal allows leakage around the piston at base of the pump head. Flow rates are low and erratic, producing chromatograms with longer than normal retention times. | Replace the piston seals (Section 5.3.13). |
| H. Broken piston; continued leaking from around the pump head after replacing the piston seal indicates a scratched or broken piston. | Replace the pistons (Section 5.3.14). |
| I. Pulse damper is leaking. Leakage may be visible at the connections to the pulse damper and/or along the seam between the two pieces of the enclosure. | 1. Pull the pulse damper out of its mounting slot.<br>2. Remove the four screws in the cover of the pulse damper and set the cover aside..<br>3. Inspect the tubing for any ruptures. If the tubing is ruptured, the pulse damper must be replaced. The tubing in the pulse damper is unique; do not attempt to substitute other tubing in its place.<br>4. If the tubing is undamaged, tighten all the fittings. Reassemble the pulse damper and remount it in the panel. Resume normal operation. |

### 5.2.2 Pump Is Difficult To Prime or Will Not Prime

| Possible Cause | Possible Remedy |
|---|---|
| A. No eluent is connected | Connect the eluent reservoir to the pump inlet line. |
| B. Eluent reservoir is empty. | 1. Refill the eluent reservoir. Be sure to filter and degas all eluents before use (Section 5.3.8).<br>2. Reprime the pump (Section 4.1.3). |
| C. Priming valve on pressure transducer is closed. | Open the valve by turning the knob on the pressure transducer counterclockwise one full turn. |
| D. The Pressure *Low Limit* is on. | The *Low Limit* must be Off during priming. Turn off the system using the *Low Limit* button-switch at the left of the System Control Panel. |
| E. Eluent reservoir is not pressurized. | Connect the pneumatic line to the eluent bottle and apply 5 to 10 psi of pressure. |
| F. The possible solutions noted in A through E, above, have been tried and pump is still difficult to prime or will not prime. | 1. Check for liquid leaks at the junction between the pump heads and the pump casting. Replace the piston seals on any pump head with a leak. (Section 5.3.13).<br>2. Clean both the inlet and outlet check valves on each head (Section 5.3.12). |

### 5.2.3 Pump Loses Prime

| Possible Cause | Possible Remedy |
|---|---|
| A. Eluent Reservoir is empty. | Refill the eluent reservoir. Be sure to filter and degas all eluents before use (Section 5.3.8). |
| B. Dissolved gas in eluent. | Dionex strongly recommends degassing all eluents and using pressurized eluent reservoirs. This helps prevent bubbles from forming in the eluent selector valves and assures optimum operation of your pump. |
| C. If A or B, above, did not cause the symptoms. | Consider the alternatives listed in Section 5.3.1 |

### 5.2.4 Pump Will Not Start

| Possible Cause | Possible Remedy |
|---|---|
| A. Power switch on pump or Front Panel is off. | Turn on switch(es). |
| B. Flow rate is set to zero. | Set the flow rate to a value greater than 0.0 mL/min. |
| C. Pressure *High Limit* is not set or is set too low. | 1. Make sure there are no blockages in the eluent lines.<br>2. Set the pressure *High Limit* at least 300-400 psi above the normal system operating pressure (Section 2.3.1). |

| | |
|---|---|
| D Relay control *Select* switch is incorrectly set for desired mode of operation. | Make sure that the relay control switch is set correctly. |

### 5.2.5 Pump Stops

| Possible Cause | Possible Remedy |
|---|---|
| A. The system pressure is less than or equal to the low pressure limit of 200 psi. The Low Limit Warning LED at the bottom of the bar gauge flashes. | 1. Make sure that the eluent reservoirs are full.<br>2. Make sure that the pump is primed ( Section 4.1.3).<br>3. Check the entire flow system for leaks.<br>4. Make sure that the waste valve on the pressure transducer is closed. To close the valve, turn the knob on the pressure transducer housing clockwise. **CAUTION: Overtightening may damage the valve and the pressure transducer housing.**<br>5. Restart the pump.<br>6. If the Alarm LED remains on, call the nearest Dionex Customer Satisfaction Representative (Section 3.1). |
| B. The pressure measured by the pressure transducer is greater than or equal to the selected high pressure limit. The High Limit Warning LED at the top of the Bar Gauge Flashes. | 1. Make sure that the selected flow rate is not too high.<br>2. Make sure that there are no blockages in the fluid lines from the pump to the waste container.<br>3. Make sure that the columns are not the source of the high pressure.<br>4. Set the high pressure limit at 200-400 psi above the normal operating pressure of the system.<br>5. Restart the pump.<br>6. If the Alarm LED remains on, call the nearest Dionex Customer Satisfaction Representative (Section 3.1). |

### 5.2.6 Pressure Readout Is 0000

| Possible Cause | Possible Remedy |
|---|---|
| A. Flow rate is set to zero. | Set the flow rate to a value greater than 0.0 mL/min. |
| B. No backpressure load. | Check the system for leaks (Section 5.3.1). If no leaks are found and the Readout remains 0000, call the nearest DIONEX Customer Satisfaction Representative (Section 3.1). |

### 5.2.7 Leaks from Pump Housing

| Possible Cause | Possible Remedy |
|---|---|
| A. A liquid line connection is loose. | 1. Make sure that the connections into the check valves are tight. Tighten fittings finger-tight, then an additional 1/8 turn (30 in-oz torque). Tighten further only if the leak continues. **DO NOT OVERTIGHTEN!**<br>2. Replace any damaged fittings. Section 5.3.1 contains fitting installation instructions for 0.125-inch OD tubing. For ferrule fitting installation instructions, refer to Dionex Manual 034213 included in the DX-100 Ship Kit. |
| B. A check valve housing is loose. | Make sure that the check valve is firmly seated in the pump body. If it is not, tighten it carefully with an open end wrench just until the leak stops (25 in-lb torque). |
| C. Pump piston seal is damaged, resulting in a loss of pressure and leaks around the pump head. | Replace the seals (Section 5.3.13) |
| D. Pump head not tight against casting. | Carefully tighten the pump head mounting screws just until the leak stops (12 in-lb torque). **DO NOT OVERTIGHTEN!** |

### 5.2.8 Pressure Transducer Leaks

| Possible Cause | Possible Remedy |
|---|---|
| A. A liquid line connection is loose. | 1. Make sure that the connections into the check valves are tight. Tighten fittings finger-tight, then an additional 1/8 turn (30 in.-oz. torque). Tighten further only if the leak continues. **DO NOT OVERTIGHTEN!**<br>2. Replace any damaged fittings (Section 5.3.1). contains fitting installation instructions for 0.125- inch OD tubing. For ferrule fitting installation instructions, refer to Dionex Manual 034213 included in the DX-100 Ship Kit. |
| B. Priming valve is open. | Close the valve by turning the pressure transducer knob clockwise just until tight, **DO NOT OVERTIGHTEN!** |

| | |
|---|---|
| C. Mounting screws are loose. | 1. Remove the valve from the pressure transducer housing by turning the knob counterclockwise until it comes loose from the housing.<br>2. Carefully tighten the four screws holding the transducer to the pump casting. **CAUTION: OVERTIGHTENING THESE SCREWS CAN SEVERELY DAMAGE THE PRESSURE TRANSDUCER HOUSING.**<br>3. Reinstall the valve in the housing. Turn the knob clockwise until the valve is seated. **DO NOT OVERTIGHTEN!** |
| D. The pressure transducer gasket or O-ring is damaged. | Replace the gasket and O-ring (Section 5.3.15). |
| E. The waste valve O-ring is damaged. | Replace the O-ring (Section 5.3.16). |
| F. The pressure transducer housing is damaged. | Replace the transducer gasket, O-ring, and housing (Section 5.3.15). |

### 5.2.9 No System Pressure

| Possible Cause | Possible Remedy |
|---|---|
| A. Pump is not primed. | Prime the pump. |
| B. Flow rate is set to zero. | Set the flow rate to a value greater than 0.0 mL/min. |
| C. No backpressure load. | Check the flow system for leaks (Section 5.3.3). |
| D. Pump piston is broken. | Replace the piston (P/N 014203) (Section 5.3.14). |

### 5.2.10 Excessive System Backpressure

| Possible Cause | Possible Remedy |
|---|---|
| A. Restriction in the hydraulic system. | Check all liquid lines and valves for crimping or blocking (Section 5.3.2). Make sure the ferrule fittings are not overtightened onto the Tefzel tubing. If necessary, refer to the Ferrule Fittings manual (Doc. 032036). |
| B. Flow rate through the columns is too High. | Reduce the flow rate. |
| C. Clogged column bed supports. | Replace the bed supports as instructed in the column manual. |
| D. Columns are contaminated. | Clean the columns (Technical Note 2R, Doc. 032036). |
| E. A blockage in the membrane suppressor or a contaminated suppressor membrane. | Refer to the membrane suppressor manual for troubleshooting procedures. |

### 5.2.11 Inoperative Valve

| Possible Cause | Possible Remedy |
|---|---|
| A. Air is not reaching valve. | Ensure that the air supply is turned on and is supplying 0.69 - 0.83 MPa (100 - 120 psi). |
| B. Air tubing harness is crimped or pinched. | Check the air tubing for blockage. Remove and replace any blocked tubing (Section 5.3.5). Reposition crimped or pinched tubes. |
| C. Air leak (air leaks are usually audible). | Isolate and eliminate leaks (Section 5.3.5). |
| D. Valve is incorrectly plumbed. | Check that the system is correctly plumbed. |
| E. Pressure bolt(s) on the injection valve are too tight. | Follow the procedure below to loosen the pressure bolts if the injection valve is plumbed into the system.<br>1. Turn off the liquid flow.<br>2. Loosen each pressure bolt slightly.<br>3. Tighten each pressure bolt finger-tight, then use an open end wrench to tighten the bolt an additional 1/8 turn. NOTE: For optimum leak-free operation, all eight pressure bolts must be evenly tightened against the internal slider.<br>4. Turn on the liquid flow and check for leaks. Tighten pressure bolts further if leaks are present. NOTE: A torque wrench can be used to evenly tighten the pressure bolts. For torque values, see the valve manual (Doc. 032678) from the Ship Kit. |

### 5.2.12 Air Leaks from Valve

| Possible Cause | Possible Remedy |
|---|---|
| Valve needs servicing. | Locate and repair the leak (Section 5.3.5). |

### 5.2.13 Peak Ghosting

| Possible Cause | Possible Remedy |
|---|---|
| A. Insufficient time between sample injections. | Wait until the previous sample has been completely eluted before making another injection. |
| B. Insufficient flush between samples. | Flush the sample loop with at least 10 loop volumes of deionized water or sample between sample injections. |

### 5.2.14 Non-Reproducible Peak Height and/or Retention Time

| Possible Cause | Possible Remedy |
|---|---|
| A. Insufficient time between sample injections. | Wait until the previous sample has been completely eluted before making another injection. |
| B. Insufficient flush between samples. | Flush the sample loop with at least 10 loop volumes of deionized water or sample between sample injections. |
| C. Column overloading. | 1. Change to a sample loop with a smaller volume.<br>2. Dilute the sample. |
| D. Liquid leaks. | Refer to Sections 5.3.3 to locate and eliminate leaks. |
| E. Compressed Air instead of nitrogen | Use nitrogen. See Section 3.4.1. |

### 5.2.15 Abnormal Retention Time and/or Selectivity

| Possible Cause | Possible Remedy |
|---|---|
| A. System is not equilibrated following an eluent change. | Allow the system to equilibrate with at least 20 column volumes of eluent before beginning an analysis. (For example, 30 minutes at 3.0 mL/min for anion separator columns or 2 hours at 0.7 mL/min for ICE separator columns). |
| B. Flow rate through system is incorrect. | 1. Check that the correct flow rate is selected.<br>2. Locate and eliminate liquid leaks (Section 5.3.3). |
| C. Contaminated or incorrect eluent. | Remake the eluent, using reagent grade chemicals and ASTM filtered Type I (18 megohm or 0.09 µS) grade, or better, deionized water. |
| D. Contaminated or degraded sample. | Take appropriate steps to prevent contamination and degradation when preparing and storing samples. |
| E. Column is contaminated. | 1. Clean the column (Technical Note 2R, Doc. 032036).<br>2. If cleaning is unsuccessful, replace the column. |

### 5.2.16 Excessive Air Consumption

| Possible Cause | Possible Remedy |
|---|---|
| Air leaks. | Locate and repair air leak (Section 5.3.5). |

### 5.2.17 No Detector Response

| Possible Cause | Possible Remedy |
|---|---|
| A. Cell not connected. | Check that cell cables are connected to panel connections. |
| B. The selected *Range* is too high; peaks are too small to measure. | Select a lower, more sensitive *Range* setting. |

| | |
|---|---|
| C. The recorder cable is disconnected; the conductivity display indicates a response, but no recorder response is observed. | Make sure the connectors on the recorder cable are pushed firmly into the recorder jacks on the rear panel (Section 3.4). |
| D. The selected *Range* is too low; the conductivity display shows only 138 with no digits to the right. | Select a higher, less sensitive *Range* setting. |

### 5.2.18 Low Detector Output

| Possible Cause | Possible Remedy |
|---|---|
| A. Selected *Range* is too high; peaks are too small to measure. | Select a lower, more sensitive *Range* setting. |
| B. Insufficient sample injected. | Increase the injection size or concentration. Section 5.3.3 describes changing the sample loop size. |
| C. Detector cell is out of calibration. | Recalibrate the cell (Section 5.3.11). |

### 5.2.19 High Detector Output

| Possible Cause | Possible Remedy |
|---|---|
| A. The selected *Range* is too low. | Select a higher, less sensitive *Range* setting. |
| B. *Auto Offset* is not set. | Press *Auto Offset* button to enable Auto Offset |
| C. The Membrane Suppressor is incorrectly installed. | Check the suppressor manual for installation in-instructions. |
| D. Sample concentration is too high. | Dilute the sample or install a smaller injection loop. (Section 5.3.2). |
| E. Wrong eluent. | Make sure that the correct eluent is used. |

### 5.2.20 Noisy Baseline

| Possible Cause | Possible Remedy |
|---|---|
| A. Air trapped in the cell; regular pulses in baseline. | Remove trapped air (Section 5.3.9). |
| B. There is a flow system leak ahead of the cell; the baseline is erratic. | Check the system for leaks (Section 5.3.3). |
| C. The operating conditions for the Membrane Suppressor are inappropriate. | Operate the suppressor as described in the suppressor manual. |

| | |
|---|---|
| D. The baseline has not been reset following a change in the Temperature Compensation setting, causing a step change in the baseline. | Whenever the Temperature Compensation DIP Switch setting is changed, reset the baseline by resetting *Auto Offset*. |

### 5.2.21 Drifting Baseline

| Possible Cause | Possible Remedy |
|---|---|
| A. Insufficient system equilibration following any changes to the operating parameters; especially apparent when operating at high sensitivities. | Allow longer system equilibration times before beginning operation. |
| B. The cell thermistor cable is not attached to the Cell Thermistor connector on the Pneumatics Control Panel. | Connect the cell thermistor cable to the Cell Thermistor connector. |
| C. Inappropriate Temperature Compensation setting. | Optimize the selected setting (Section 4.1.7). |
| D. The operating conditions for the Membrane Suppressor are inappropriate. | Operate the suppressor as instructed in the Suppressor manual. |

## 5.3 SERVICE

This section describes service and repair procedures for the DX-100. Before repairing or replacing any part, consult Section 5.2 to isolate the source of the problem.

When ordering replacement parts, please include the serial number of your DX-100 and the part numbers and quantities of the parts you want.

### 5.3.1 Tube Fittings

Two types of tube fittings are used in the DX-100. The Injection Valve and some columns are plumbed with black PEEK tubing and Dionex ferrule fittings. The rest of the system is plumbed with Dionex ThermoFlare fittings.

Instructions for installing ferrule fittings on PEEK or Tefzel tubing are found in the Ferrule Fitting Installation Manual (Doc. 034213) found in the DX-100 Ship Kit.

Instructions for installing and making ThermoFlare fittings are given in the following paragraphs.

### 5.3.1.1 Tightening Dionex ThermoFlare Fittings

Dionex ThermoFlare fittings are used for tubing connections made with 0.3-mm (0.012-inch) and 0.5-mm (0.020-inch) ID Tefzel tubing. When a leak occurs, tighten the fitting by approximately 1/8 turn with a 5/16-inch wrench. If the leak persists, remake the flare or replace the tubing.

**WARNING: Do not try to remake the flare after the tubing has been used with acetonitrile, tetrahydrofuran, or methanol. You will not be able to obtain a good sealing surface once the tubing has been used with any of these solvents.**

### 5.3.1.2 Ordering Tubing with Factory-Made ThermoFlares

If you prefer not to make your own flares, tubing with factory-made flares is available from Dionex in the following IDs and lengths:

**0.3-mm (0-.012-inch) ID Tubing - White**

10 cm (4 inches) long (P/N 039335)
20 cm (8 inches) long (P/N 039336)
30 cm (12 inches) long (P/N 039337)
60 cm (24 inches) long (P/N 039338)

**0.5-mm (0.020-inch) ID Tubing - Blue**

10 cm (4 inches) long (P/N 039339)
40 cm (16 inches) long (P/N 039340)
90 cm (36 inches) long (P/N 039343)
180 cm (72 inches) long (P/N 039341)

### 5.3.1.3 Making and Installing Thermoflare Fittings

The sealing surface of a fitting is made by first heating and then flaring the end of the tubing. A special flaring tool (120 VAC version, P/N 037196; 230 VAC version, P/N 037197) is required to make ThermoFlares. A manual (Doc 032843) explaining how to make flares is shipped with the tool. The procedure for reflaring and reinstalling tubing is given below.

1. Slide the existing fitting up the tubing to expose the old flare.

2. Use a sharp razor blade to cut off the old flare. Make the cut so that the new surface is cleanly perpendicular to the sides of the tubing.

3. Inspect the existing fitting for damage (e.g., stripped threads) and replace it if necessary with a new fitting (P/N 030540). Slide the fitting and a washer over the cut end of the tubing.

4. Loosen the clamp screws on the flaring tool and slide the cut end of the tube through the tool until it reaches the other side of the slot. Tighten the clamp screws firmly.

5. Turn the flaring knob clockwise into the tubing until it bottoms out, flaring out the end of the tubing.

6. Loosen the clamp screws and remove the tubing. Inspect the flare to make sure that it is concentric with the tubing axis.

7. The fitting is ready for use. Tighten fittings finger-tight, then an additional 1/8 turn (30 in/oz). Tighten further only if a leak occurs.

   **NOTE**: For best results install a prepared fitting within five minutes of making the flare. If left longer, the tubing tends to relax back to its original shape, making the flare unusable.

### 5.3.2 Changing the Sample Loop

Peak response is directly related to the concentration of sample and the volume injected. The sample volume may be changed by changing the volume of the injection valve sample loop. With most samples, use of a sample loop larger than 100 µL may result in column overloading and non-linear response. This overloading volume may be even smaller with some types of sample.

1. Turn off the pump.

2. Open the DX-100 door.

3. Disconnect the sample loop from ports 2 and 6 on the injection valve (refer to Figure 5-1 for port identification). Use a 1/2-inch wrench to prevent the valve pressure bolts from turning when removing the fittings.

4. Prepare a new sample loop from 0.3 mm ID tubing (P/N 035548). Allow 1.37 cm (0.54 inches) of tubing, including the fittings, for each microliter of loop volume. When making sample loops of 100 µL or more, use 0.5 mm ID tubing (P/N 035864). Allow 49 cm of tubing including fittings, for each 100 µL. **NOTE**: Sample loops of 10, 25, 50, 100, 200, 250, 500, and 1000 µL (P/Ns 036104, 036105, 036106,, 030391, 036107, 030392, 030393, and 036108, respectively) are available from Dionex.

5. Install the new sample loop between ports 2 and 6 on the injection valve. Use a wrench to prevent the pressure bolt from turning when tightening the fittings. Tighten the fittings finger-tight, then an additional 1/8 turn. Tighten further only if a leak appears. **DO NOT OVERTIGHTEN!**

### 5.3.3 Isolating and Eliminating a Restriction in the Hydraulic System

A restriction in the hydraulic system causes excessive system backpressure.

1. Begin pumping eluent through the system (including the columns) at the flow rate normally used.

2. Following the hydraulic schematic (Figure 2-2 or 2-3), work backward through the system, beginning at the cell exit. One at a time, loosen each fitting and observe the pressure. The connection at which the pressure drops indicates the point of restriction.

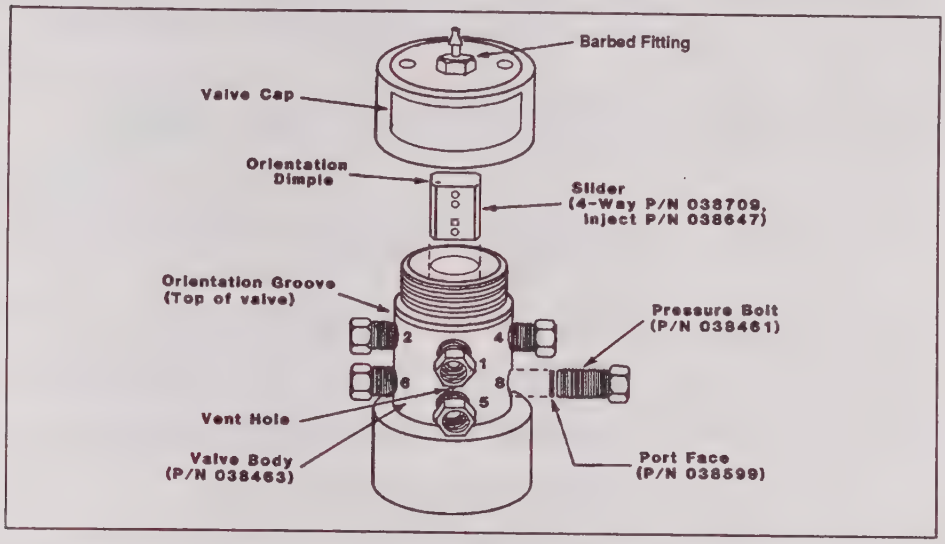
3. Remove the restriction either by flushing or by replacing the section of tubing.

4. If the restriction is caused by the injection valve, disassemble the valve and clean the slider (Section 5.3.6). If cleaning the valve slider does not eliminate the backpressure problem, replace the port face (Section 5.3.7).

## 5.3.4 Isolating and Eliminating Injection Valve Liquid Leaks

A poor seal between the fitting sealing surface and the valve pressure bolt, or between the port face (Figure 5-1) and the valve slider may cause the injection valve to leak. The flow rate through the system may be low, producing chromatograms with longer than normal retention times, and reproducibility may be poor.

**Figure 5-1**
**DX-100 Injection Valve**




1. Tighten any loose fittings finger-tight, then an additional 1/8 turn. Tighten further only if a leak continues. DO NOT OVERTIGHTEN. Use an open end wrench to prevent valve pressure bolts from turning when tightening connections to the valves.

2. If tightening the fitting does not stop the leak, disconnect the fitting from the valve. Occasionally, fittings stop leaking if simply disconnected and then immediately reconnected. If the leak continues, install a short length of tubing, with fittings, between the pressure bolt and the suspect fitting. Connect the fitting to the tubing with a union. A leaking fitting also leaks inside the union. If necessary, replace the fitting.

   **NOTE: When connecting or disconnecting fittings from the valve pressure bolts, use a wrench to prevent the nut from turning.**

3. Make sure all valve pressure bolts are evenly tightened. For torque requirements, refer to the valve manual (Document No. 032678) from the Ship Kit.

   **CAUTION: Overtightening may break the bolt off in the valve body.**

4. If tightening the pressure bolts does not stop the leak, refer to Section 5.3.6.

## 5.3.5 Correcting Injection Valve Air Leaks

An air leak around the valve piston or cap results in excessive air consumption and may cause the valve to operate sluggishly or not at all.

1. Remove the injection valve from its mounting clip.

2. Make sure that the top and bottom caps are tight. If necessary, tighten them finger-tight.

3. If the leak continues, determine which end of the valve leaks by activating the corresponding switch on the Pneumatic Control Panel. Air will escape through the vent hole when the leaking end is pressurized.

4. Disconnect the air line from the leaking cap. If necessary, use the valve switch to turn off the air through the disconnected line.

5. Unscrew the leaking cap from the valve body (Figure 5-1). Inspect the cap O-ring (P/N 035970) for cracks and replace if necessary.

6. Press the disconnected air line (Step 4) against the small vent hole between ports 1 and 5. Activate the valve switch to apply air pressure against the slider. The piston will pop out of the valve body.

7. Inspect the piston O-ring (P/N 035925) and replace it if necessary.

8. Carefully lubricate the piston O-ring with a small amount of silicone grease.

   **CAUTION: Use the grease sparingly. Excess grease may contaminate the eluent.**

9. Press the piston back into the valve body.

10. Make sure that the cap O-ring is inside the cap. Screw the cap back onto the valve body. Tighten it only finger-tight.

11. Reconnect the air line to the valve.

### 5.3.6 Cleaning the Injection Valve Slider

A dirty or plugged valve slider causes excessive system backpressure and may cause poor chromatographic performance.

1. Turn off the pump and the eluent selector valves.

2. Remove the plugged valve from its mounting clip.

3. Disconnect the air line from one cap. If necessary, use the valve switch to turn off the air through the disconnected line.

4. Unscrew one cap from the valve body. Be careful not to lose the cap O-ring.

5. Press the disconnected air line (Step 3) against the small vent hole between ports 1 and 5. Activate the valve switch to apply air pressure against the slider. The piston will pop out of the valve body.

6. Turn off the air supply.

7. Carefully loosen each of the pressure bolts 1/8 turn to disengage the port faces from the slider. Do not loosen any further.

8. Remove the other cap. Push on the slider to force the other piston out of the valve body.

9. Carefully push the slider out of the valve body with a blunt rod.

10. Inspect the slider surface. If there are scratches in the surface, replace the slider (P/N 030340).

11. Place the slider in a small container of deionized water or methanol. Sonicate or agitate vigorously for several minutes.

12. Rinse the slider with deionized water. Blow any water out of the holes and inspect them for blockage. Use a fine piece of wire (0.025 inch) to dislodge any remaining blockage. Be careful not to scratch the slider surface. If the blockage cannot be removed, replace the slider.

13. Push the slider back into the valve body. Orient the dimple on the top of the slider between ports 1 and 2 of the valve body (Figure 5-1).

    **The valve operates properly only if the slider is correctly oriented in the valve body. The groove around the valve body indicates the top of the valve body. The small vent hole is between ports 1 and 5. The ports are numbered clockwise from the top around the valve body. The slider connects the ports as follows:**

    | | |
    |---|---|
    | *Sample* Switch in LOAD position (slider down): | 1 → 4 and 2 → 3 |
    | | 5 → 8 and 6 → 7 |
    | *Sample* Switch in INJECT position (slider up): | 1 → 2 |
    | | 5 → 6 |

14. Inspect the piston O-rings (P/N 035925) and replace them if necessary.

15. Carefully lubricate the piston O-rings with a small amount of silicone grease and re-assemble the valve (Section 5.3.4).

16. Tighten each pressure bolt finger-tight, then use an open end wrench to tighten an additional 1/8 turn. For optimum leak-free operation, all eight pressure bolts must be evenly tightened against the slider (12 in.-oz.). A special torque wrench kit (P/N 038943) is available from your Dionex Customer Satisfaction Representative.

17. Turn on the liquid flow and check for leaks. Tighten pressure bolts further if leaks appear.

    **CAUTION: Overtightening may break off the bolt in the valve body.**

18. See Section 5.3.7 if cleaning the valve slider does not eliminate the backpressure problem.

### 5.3.7 Replacing a Damaged Port Face

A scratched port face may cause a leak around the pressure bolt. A plugged port face will cause excessive system backpressure and may cause poor chromatographic performance.

1. Turn off the liquid flow.

2. Remove the valve from its mounting clip.

3. Disconnect the tube fitting from the suspect pressure bolt, then unscrew the pressure bolt from the valve body.

4. Inspect the port face. If it is scratched or plugged, carefully pry it out of the pressure bolt. Press a new port face (P/N 035909) into the pressure bolt. **DO NOT SCRATCH THE SEALING SURFACES!**

5. Screw the pressure bolt into the valve body. Tighten each pressure bolt finger-tight, then use an open end wrench to tighten an additional 1/8 turn. For optimum leak-free operation at 4000 psi, all eight pressure bolts must be evenly tightened against the slider (128 in.-oz.). A special torque wrench kit (P/N 038943) is available from the Dionex Service Department. A lower torque value may be used with a lower operating pressure. Valve life will be extended by using the lowest torque value which provides a secure seal.

6. Turn on the liquid flow and check for leaks. Tighten pressure bolts further if leaks appear.

   **NOTE:** For optimum leak-free operation, a torque wrench may be used to evenly tighten pressure bolts. Refer to the valve manual (Document No. 032678) for torque requirements.

   **CAUTION: Overtightening may break off the bolt in the valve body.**

7. Reconnect the tube fitting to the pressure bolt. Use a wrench to prevent the pressure bolt from turning while tightening the fitting. Tighten the fitting finger-tight, then an additional 1/8 turn. Tighten further only if leaks appear. DO NOT OVERTIGHTEN.

### 5.3.8 Degassing Eluents

When using eluents containing volatile components it may be necessary to degas the eluent prior to use. Degas the eluents daily prior to beginning operation.

1. Pour the prepared eluent into a vacuum flask.

2. Attach the flask to a water aspirator and aspirate for 5 minutes with shaking or sonication.

3. Remove the flask from the aspirator, taking care not to allow water to back-flow from the aspirator into the flask.

4. Use the degassed eluent directly from the flask or carefully pour it into an eluent reservoir. Avoid shaking.

### 5.3.9 Removing Air Trapped in Cell

Air trapped in the cell causes a regular pulsation of the baseline. The trapped air may be a result of air introduced into the column during installation, or may be the result of eluent outgassing.

1. Increase the cell backpressure by installing a length of 0.3-mm (0.012-inch) ID tubing (P/N 035548), with fittings on both ends, between the cell outlet and the waste line. Use a union (P/N 034447) to connect the tubing to the waste line. This additional piece of tubing shrinks bubbles, allowing them to pass more easily through the cell.

2. If the pulse continues, bypass all columns and valves. Connect the cell directly to the eluent pump, using a length of 0.5-mm (0.020-in) ID tubing (P/N 035519) and a union (P/N 024447). Loosen the cell exit line to allow free flow from the cell. Pump eluent through the cell at 9.9 mL/min. Tap the cell body firmly (e.g., with a small wrench) to dislodge any air bubbles. Continue until the pulse disappears. Reduce the flow rate to the rate required for your application and tighten the cell outlet fitting. CAUTION: Do not pump eluent through the columns at flow rates greater than 4.0 ml/min. System over-pressuring and column damage may result.

3. Avoid pumping air through the columns and cell.

### 5.3.10 Cleaning Cell Electrodes

Normally it is not necessary to clean the conductivity cell, but if fouling is suspected the cell should be cleaned. After cleaning the cell you must recalibrate it in accordance with the instruction found in Section 5.3.11 of this manual.

1. Prepare the solutions listed below. Pour the prepared solutions into labeled containers. **NOTE:** Use filtered ASTM Type I (or better) deionized water to prepare all solutions.

   a. 3 M $HNO_3$ cleaning solution: dilute 200 mL concentrated $HNO_3$ (s.g. 1.42) to one liter with deionized water.

   b. 0.01 M KCl stock solution: dissolve 0.7456 g of reagent grade KCl in one liter of deionized water.

   c. 0.001 M KCl calibration solution: dilute 100 mL of the stock solution to 1 liter with deionized water.

2. Connect the container of 0.001 M KCl to the pump.

3. Disconnect the line between the suppressor outlet and the cell inlet at the suppressor.

4. Connect a female luer adaptor (P/N 024305) to the tubing, using a union (P/N 024447).

5. Fill a 10 mL syringe (P/N 016387) with the 3 M $HNO_3$ solution. Screw the syringe into the luer adaptor.

6. Turn off the DX-100 main AC power switch (located on the right side panel).

7. Inject 5 mL $HNO_3$ through the cell.

8. After two minutes, push the remaining 5 mL of solution through the cell. Wait two minutes.

9. Fill the syringe with 10 mL of deionized water. Inject the water through the cell.

10. Turn on the DX-100 main AC power switch.

### 5.3.11 Calibrating the Cell Constant

**NOTE:** Clean the cell electrodes before calibrating the cell constant (see Section 5.3.10).

1. Disconnect the pump eluent line from the injection valve.

2. Disconnect the syringe and luer adaptor from the union. Connect the eluent line from the pump to the union.

3. Pump the 0.001 M KCl calibration solution through the cell at 2 mL/min. After 5 minutes, reduce the flow rate to the value used during analysis and pump for an additional minute. **NOTE:** Conductivity is flow-rate sensitive. Calibrate the detector at the flow rate used in the majority of your applications.

4. Set the Detector *Range* to 300 µS.

5. Use DIP Switch 1 to set the temperature compensation to 1.7%. NOTE: The factory-set default is 1.7%; this value can be used for most applications. When measuring absolute conductivity, determine the optimum value (see Section 4.1.7) for each batch of calibration solution.

6. Disconnect one of the cell drive connections on the left side of the Pneumatic Control Panel.

7. Press the *Auto Offset* button to turn on Auto Offset. With the cell drive disconnected, this is the zero conductivity point.

8. Reconnect the cell drive.

9. Locate the Cell Gain Adjust potentiometer, visible through the small hole on the left side of the DIP Switch Panel. Continue pumping calibration solution through the cell. Use a tuning screwdriver (P/N 035617) from the Ship Kit to adjust the Cell Gain Potentiometer until the digital display value is equal to 147 (±2 µS).

10. Replace the container of 0.001 M KCl calibration solution with a container of deionized water. Pump deionized water through the liquid lines at 2.0 mL/min for at least 5 minutes to flush the calibration solution from the system.

11. Reconnect the pump to the injection valve.

12. Reconnect the liquid line from the cell inlet to the suppressor outlet.

13. Use DIP Switch 1 to reset the temperature compensation to the optimum value for the eluent in use. The detector is now calibrated and ready for use.

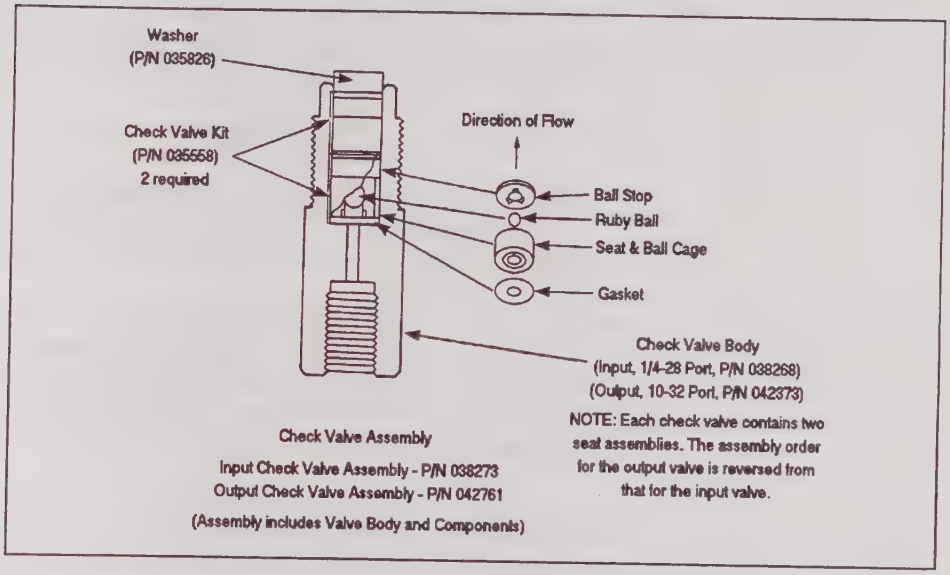
## 5.3.12 Disassembling and Cleaning Check Valves

A dirty check valve causes the flow rate to be erratic and can cause the pump to lose prime and/or be difficult to reprime.

Dionex strongly recommends replacing check valves (P/N 038273, inlet; P/N 042761, outlet) rather than cleaning them If, however, you prefer to clean them, follow the procedure below.

**CAUTION: Disassemble and reassemble check valves only under very clean conditions; minute particles on the valve parts will cause erratic pumping.**

**Figure 5-2 Check Valve Assembly (Input Valve Shown)**


Washer
(P/N 035826)
Check Valve Kit
(P/N 035558)
2 required
Direction of Flow
Ball Stop
Ruby Ball
Seat & Ball Cage
Gasket
Check Valve Body
(Input, 1/4-28 Port, P/N 038268)
(Output, 10-32 Port, P/N 042373)
NOTE: Each check valve contains two seat assemblies. The assembly order for the output valve is reversed from that for the input valve.
Check Valve Assembly
Input Check Valve Assembly - P/N 038273
Output Check Valve Assembly - P/N 042761
(Assembly includes Valve Body and Components)

1. Prime the pump (Section 4.4.2). If the pump remains unprimed, proceed to the next step.

2. Turn off the power switch on the DX-100 right side panel to prevent the pump from being inadvertently turned on. Turn off the pump switch.

3. Prepare a clean work area, a small beaker to hold the parts, and tweezers to handle them.

4. Disconnect the tube fittings from both the inlet and outlet check valve housings.

5. Using a 1/2-inch box wrench, carefully remove both check valve housings from the pump head. Mark the housings to indicate inlet or outlet.

NOTE: Although the check valves outwardly appear similar, the outlet check valve assembly has a 10-32 port, while the inlet assembly has a 1/4-28 port. Their internal assemblies are also different. The Ball Stop; Ruby Ball; Seat and Ball Cage; and Gasket, shown in Figure 5-2, are installed in reverse order in the output check valve. Keep the two check valves separated and well-identified at all times.

6. Follow the steps below to disassemble the check valve:

   a. Wrap the unthreaded end of the housing in a lint-free tissue.

   b. Carefully push a rod (maximum diameter 0.020 inch) into the internally threaded end of the check valve housing. Apply enough pressure to the rod to force the internal parts out of the housing.

   **CAUTION : Do not lose any of the internal components.**

7. Place all of the parts in a small beaker of deionized water or isopropyl alcohol. Sonicate or agitate vigorously for five minutes.

8. Carefully rinse the parts with deionized water. Handling the clean parts with tweezers, inspect each part under magnification for contamination or damage. Replace any damaged parts (check valve kit P/N 035558; washer P/N 035826).

   **NOTE:** Each check valve requires two check valve kits for complete rebuilding.

   **CAUTION: Some of the parts may float in water. Do not lose them when rinsing.**

9. Reassemble the check valve. Refer to Figure 5-2 for the correct assembly order.

   **NOTE:** The washer nearest the bottom of the check valve body is seldom removed. Do not install a new washer on top of it when reassembling the check valve. The individual parts for the inlet and outlet check valves are identical, and inter changeable. The order in which they are placed in the housing is determined by the direction of liquid flow through the check valve.

10. Verify that the check valve is correctly assembled by blowing through it in the direction of liquid flow. If the valve is correctly assembled, air will flow through it. Then blow through the check valve in the direction opposite of liquid flow. If the valve is correctly assembled, air will not flow through it.

11. Install the clean check valves in the pump head. Be sure to install the inlet check valve on the bottom of the head and the outlet check valve on the top. Tighten only enough to seat (25 in-lb torque).

    **CAUTION: Overtightening may damage the pump head or the check valve housing and crush the check valve seats.**

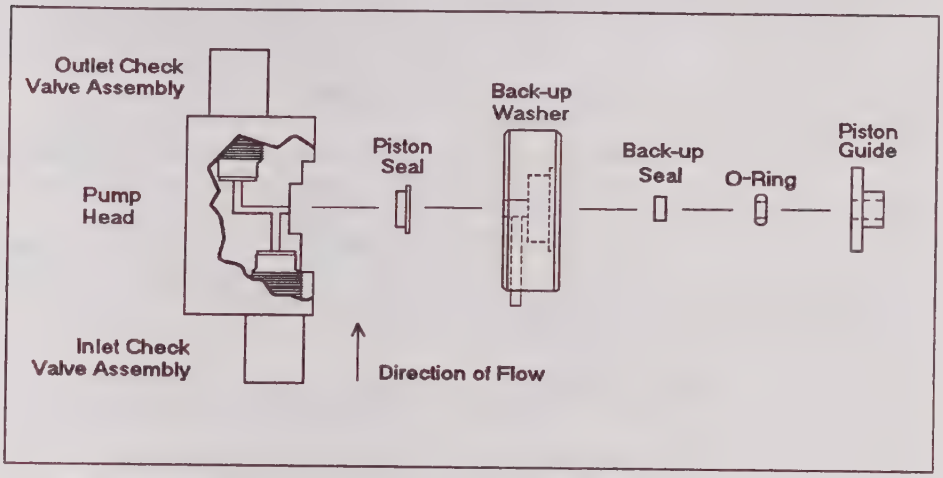
12. Reconnect the liquid lines. Turn on the power switch on the DX-100 right panel.

13. Prime the pump (Section 4.1.3).

## 5.3.13 Replacing a Piston Seal

A damaged piston seal allows leakage around the piston at the head mounting plate or around the base of the pump head. Flow rates will be low and erratic, producing chromatograms with longer than normal retention times.

Figure 5-3
Pump Head
Assembly




1. Turn off the pump.

2. Turn off the power switch on the DX-100 right side panel. This prevents the pump from being accidentally turned on, possibly breaking the piston.

3. Disconnect the tube fittings from the pressure transducer.

4. Disconnect the tube fittings from the inlet check valve.

5. Hold the head firmly against the pump housing to compensate for the spring loading, and remove the two nuts.

6. Slowly release the head, allowing it to separate from the housing. CAREFULLY disengage the head from the sapphire piston by pulling the head straight off and away from the mounting guides. Be especially careful not to snap the piston if the internal spring is stuck to the piston guide.

   **CAUTION: Lateral motion when disengaging the head from the piston can break the piston.**

7. Place the head, front end down, on a clean work area. Lift the piston guide and back-up washer off, exposing the seal (Figures 5-3 and 5-4).

8. Remove the piston seal from the head. Discard the seal.

9. Remove the O-ring and back-up seal from the back-up washer.

10. Carefully push the new piston seal (P/N 035686) into the head. When properly installed, the piston seal is almost flush with the indented surface of the head.

11. Press a new back-up seal (P/N 036901) into the O-ring (P/N 035776). If necessary, also replace the O-ring. Then press the O-ring and back-up seal into the back-up washer.

12. Press the back-up washer into the head, followed by the piston guide.

13. Remove the spring, spring guide, spring retainer, and piston from the pump housing (Section 5.4.13). Inspect the interior of the housing for liquid and corrosion. Clean up any spills and carefully clean any signs of corrosion from the piston and the interior of the housing. Reinstall the piston.

14. Carefully slide the pump head straight onto the mounting guides. Apply gentle pressure to push the piston through the seal. Guide the spring onto the piston guide.

    **CAUTION: Avoid all lateral motion when sliding the head onto the piston. Failure to slide the head straight on will damage the piston seal and back-up seal, as well as break the piston.**

15. Hold the head firmly against the pump housing and replace the two nuts. Use a wrench to tighten them evenly (12 in-lb torque).

16. Reconnect the liquid line to the inlet check valve.

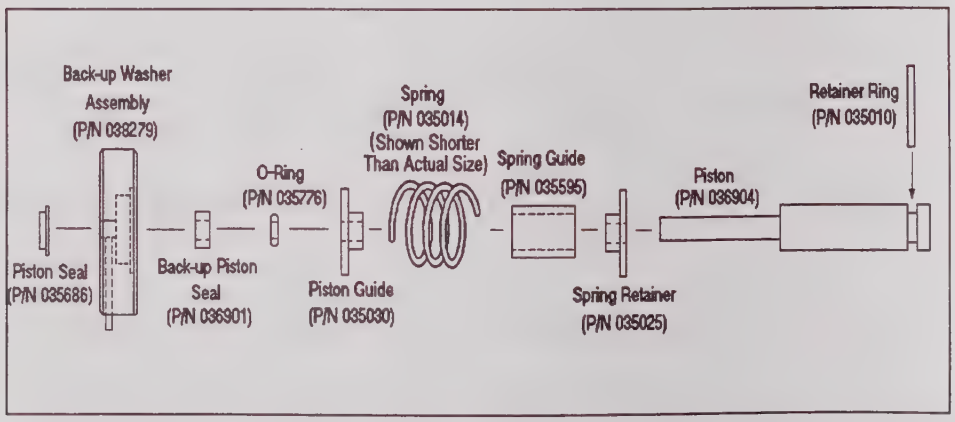
17. Reconnect the tube fittings to the pressure transducer.

18. Turn on the POWER switch on the right side panel. Prime the pump as instructed in Section 4.1.3.

## 5.3.14 Replacing a Pump Piston

Continued leaking from around the pump head after replacing the piston seal indicates a scratched or broken piston.

**Figure 5-4 Piston Pump Assembly**


Back-up Washer Assembly (P/N 038279)
Spring (P/N 035014) (Shown Shorter Than Actual Size)
Retainer Ring (P/N 035010)
O-Ring (P/N 035776)
Spring Guide (P/N 035595)
Piston (P/N 036904)
Piston Seal (P/N 035686)
Back-up Piston Seal (P/N 036901)
Piston Guide (P/N 035030)
Spring Retainer (P/N 035025)

1. Turn off the power switch on the side panel. This prevents the pump from being accidentally turned on, which could break the piston.

2. Disconnect the tube fittings from the pressure transducer.

3. Disconnect the tube fittings from the inlet check valve.

4. Hold the head firmly against the pump housing to compensate for the spring loading and remove the two nuts.

5. Slowly release the head and allow it to separate from the housing. CAREFULLY disengage the head from the sapphire piston by pulling the head straight off and away from the mounting guides. Be especially careful not to snap the piston if the spring is stuck to the piston guide.

   **CAUTION: Lateral motion when disengaging the head from the piston will break the piston.**

6. Remove the spring, spring guide, spring retainer, and piston by pulling them straight out, away from the pump housing (Figure 5-4).

7. If the piston is broken, replace the piston seal and the back-up seal (Section 5.3.13). This will prevent pieces of broken piston from scratching the new piston assembly.

8. Remove the retainer ring (P/N 035010) from the old piston assembly and install it on the new piston assembly (P/N 036904).

9. Carefully slide the spring retainer onto the piston assembly.

10. Slide the assembled piston back into the piston housing.

11. Slide the spring over the piston, positioning it flush against the spring retainer.

12. Carefully slide the pump head straight onto the alignment rods. Gently push the piston through the seal. Guide the spring over the piston guide.

    **CAUTION:** Avoid all lateral motion when sliding the head onto the piston. Failure to slide the head straight over the piston will damage the piston seal and back-up seal, as well as break the piston.

13. Hold the head firmly against the pump housing and replace the two nuts. Use a wrench to tighten them evenly (12 in-lb torque).

14. Reconnect the liquid lines to the check valve housing.

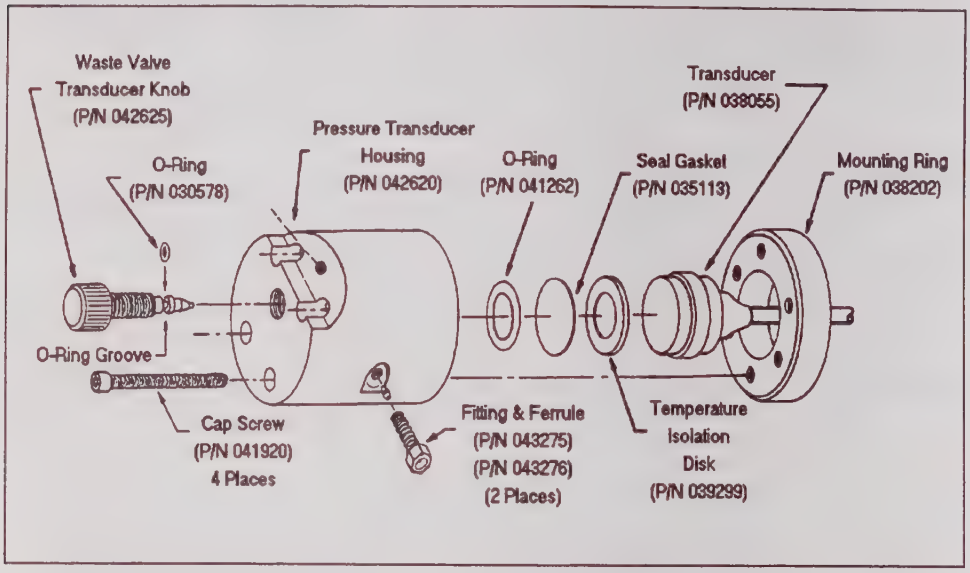
15. Reconnect the tube fittings to the pressure transducer.

16. Turn on the power switch on the side panel. Prime the pump as instructed in Section 4.1.3.

## 5.3.15 Replacing the Pressure Transducer Gasket and O-Ring

A damaged gasket or O-ring causes leakage around the base of the pressure transducer housing at the mounting ring. Flow rates will be low, resulting in chromatograms with excessively long retention times.

Figure 5-5
DX-100 Pressure Transducer




1. Press the *Pump* button-switch to turn off the pump.

2. Disconnect the liquid lines between the pressure transducer housing and the check valve housings.

3. Disconnect the waste line from the pressure transducer housing.

4. Remove the output line from the pressure transducer housing.

5. While holding the housing firmly, remove the four screws holding the housing to the mounting ring.

6. Gently pull the housing away from the ring, exposing the pressure transducer face (Figure 5-5).

7. Remove the temperature isolation disk, the gasket, and the O-ring from the housing.

8. Install a new O-ring (P/N 041262) and gasket (P/N 035113) in the housing. Place the temperature isolation disk on top of the gasket.

9. Reposition the housing on the ring and replace the screws. Carefully tighten the screws until evenly tight (20 in-lb).

   **CAUTION: Overtightening or uneven tightening may damage the pressure transducer housing.**

10. Reconnect the liquid lines from the check valve housings. Reconnect the outlet line and the waste line.

### 5.3.16 Replacing the Pressure Transducer Waste Valve O-Ring

A damaged O-ring causes leakage around the base of the pressure transducer waste valve knob. Flow rates will be low, resulting in chromatograms with longer retention times.

1. Press the *Pump* switch on the System Control Panel to turn off the pump.

2. Remove the valve from the pressure transducer housing by turning the knob counter-clockwise until it comes loose from the housing (Figure 5-5).

3. Cut through the O-ring with a razor blade, being careful not to scratch the valve body. Remove the O-ring.

4. Carefully slide a new O-ring (P/N 035776) over the end of the valve and push it into the groove.

5. Wipe a very thin film of silicone grease on the O-ring.

   **CAUTION:** Excessive grease may contaminate the eluent as it flows through the pressure transducer housing.

6. Reinstall the valve in the housing, turning the knob clock wise until the valve is seated.

   **CAUTION:** Overtightening the valve may damage the seat and pressure transducer housing.

# APPENDIX A
# Principles of Conductivity Detection

## How Conductivity is Measured

The conductivity of a solution is measured by applying an alternating voltage between two electrodes in a conductivity cell. At any instant in time, negatively charged anions migrate toward the positive electrode and positively charged cations migrate toward the negative electrode. The inverse of the measured solution resistance is the *conductance*, measured in siemens (S). The conductivity of the the solution is the conductance which would be measured in a standard cell containing electrodes of 1 $cm^2$ surface area held 1 cm apart. The units of conductivity are siemens per cm (S/cm).

## Conductivity of Solutions

The conductivity of a dilute solution is the sum of the individual contributions to conductivity of all the ions in the solution multiplied by their concentration; i.e., conductivity is directly proportional to concentration. This is called Kohlraush's law of independent migration. It states that each ion carries its portion of the total conductivity without being affected by any of the other ions in solution. Stated as an equation:

$$\kappa = \frac{\Sigma_i \lambda_i^\circ c_i}{1000}$$

where $\kappa$ is the measured conductivity in S/cm, and $c_i$ is the concentration of the ions in equivalents/L. (Equivalents/L equals moles/L times the charge on the ion.) $\lambda_i^\circ$, the ionic limiting equivalent conductivity, is specific for each ion. It is the conductivity of the ion divided by concentration and extrapolated to infinite dilution. Table A-1 lists limiting equivalent conductivities for a number of organic and inorganic ions. The unit for $\lambda_i^\circ$ is $S \times cm^2$ / equiv.

**Equivalent Conductivities in Aquaeous Solution, 25 C**
**($S \times cm^2$ / equiv)**

| Anions | $\lambda^\circ$ | Cations | $\lambda^\circ$ |
|---|---|---|---|
| $OH^-$ | 198 | $H^+$ | 350 |
| $F^-$ | 54 | $Li^+$ | 39 |
| $Cl^-$ | 76 | $Na^+$ | 50 |
| $Br^-$ | 78 | $K^+$ | 74 |
| $I^-$ | 77 | $NH_4^+$ | 73 |
| $NO_3^-$ | 71 | $Mg^{2+}$ | 53 |
| $HCO_3^-$ | 45 | $Ca^{2+}$ | 60 |
| $SO_4^{2-}$ | 80 | $Sr^{2+}$ | 59 |
| $Acetate^-$ | 41 | $CH_3NH_3^+$ | 58 |
| $Benzoate^-$ | 32 | $N(CH_3CH_2)_4^+$ | 33 |

Values of $\lambda°_i$ from Table A-1 can be used to calculate the conductivities of solutions containing ions. For example, the limiting equivalent conductivity at 25 C for NaCl is the sum of the ionic limiting equivalent conductivity for $Na^+$, 50.1; plus that of $Cl^-$, 76.4; or 126.5. A 0.1 mM solution of NaCl at 25 C will have a conductivity of 0.1 × 126.5, or 12.65 µS/cm. So far, only dilute solutions have been discussed. As concentration increases, the direct proportionality between conductivity and concentration is lost. However, at the analyte concentrations normally encountered in ion chromatography (below 1 millimolar), conductivity is generally proportional to concentration. For example, the equivalent conductivity at 25 C of KCl at infinite dilution is 149.9; and at 1 mM, 146.9, a decrease of only 2%. (The conductivity of an eluting analyte cannot be assumed to be directly proportional to concentration, because ionic components of the mobile phase may be contained in the eluting volume.)

If the electrolyte is a weak electrolyte such as an acid or base with only partial dissociation, then $c_i$ must be replaced by the concentration of the dissociated ions only, since only they contribute to conductivity. For acids and bases, the pK values and the solution pH can be used to calculate the extent of dissociation. Since the dissociation of a weak acid or base is also dependent on its concentration, calibration curves where the analyte is a weak acid or base may not be linear.

## Effect of Solvent on Conductivity

The mobility of ions is affected by the solvent, with the principle effects being changes in ion solvation and solvent viscosity. Ions are more mobile in solvents of lower viscosity. It is not necessary to know values such as the extent of solvation and viscosity, since quantitative analysis is performed by comparing the conductivity of the analyte in the sample to the conductivity of the same analyte from a standard (or standards).

## Effect of Temperature on Conductivity

Mobility, and therefore conductivity, is greatly affected by temperature. The conductivity of an aqueous solution is found experimentally to rise a little less than 2% per degree C. (This dependence is described in a complex equation developed by Onsager.) Therefore, it is necessary to correct the measured conductivity to a constant temperature, or errors in concentration measurements could result as the eluent temperature changes. The DX-100 accomplishes this by correcting the measured conductivity to that which would be measured at 25 C, by measuring the cell temperature with a thermistor and multiplying the conductivity by a temperature dependent constant. This constant is called the *temperature compensation factor*, and is expressed in units of percent per degree C. Use the procedure detailed in Paragraph 4.1.7 to set the temperature compensation factor.

## Chemical Suppression

### Principals

Species detected by conductivity are by their nature ionic, so ion-exchange and ion-pair chromatography are by far the most commonly used separation methods. These methods require eluents containing strong electrolytes. This causes a problem: how to detect the ionic analytes without the detector being overwhelmed by the ions in the eluent. The best method of solving this problem is by neutralizing the eluent in a suppressor.

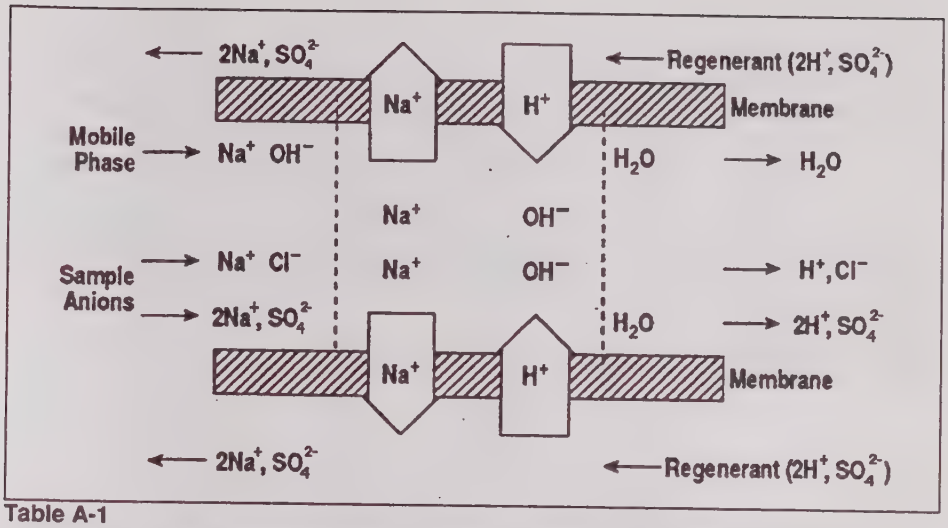



**Table A-1**

**Schematic of chemical suppression (anion exchange). Sodium counterions from the eluant cross the cation-exchange membranes and are replaced by hydrogen ions from the sulfuric acid regenerant, neutralizing the highly conductive hydroxide to water and greatly increasing the signal-to-noise ratio.**

The mechanism of suppression, using sodium hydroxide as the eluent for anion-exchange separation, is illustrated in Figure A-1. Analyte anions elute from the column with sodium counterions. They enter the suppressor between two cation-exchange membranes. On the other side of these membranes a dilute solution of sulfuric acid (the regenerant) flows in the opposite direction. The membranes have fixed negative sulfonic acid functional groups and therefore exclude anions. Positive ions can pass through the membrane freely. Therefore, sodium ions from the eluent flow across the membranes into the regenerant side. An equal number of hydrogen ions from the regenerant flow across the membrane and enter the eluent side, thus neutralizing the highly conductive hydroxide to water. The resulting background conductivity is near zero, considerably lower than before suppression. Also, the counterion to the anion analytes is now hydrogen ion, which has seven times higher conductivity than the original sodium counterion. Since both the anion analyte and the cation counterion produce the response at the detector, the response is increased. The suppressor both lowers the background conductivity (and therefore the baseline noise and drift) and increases the signal. Continuous recycling of regenerant through a large ion exchange column (provided by the Dionex AutoRegen Accessory) allows the suppression system to be used unattended for weeks.

For cation ion chromatography, the suppressor membranes are anion exchange polymers. They allow anions to pass freely but exclude cations. Dilute acids such as hydrochloric acid are used in the eluent. In the suppressor, the chloride counterions are replaced by hydroxide, neutralizing the acidic eluent and providing the highly conductive hydroxide counterion to the analyte cations. Tetrabutylammonium hydroxide is used as the regenerant because it is excluded from the anion exchange membranes.

### Advantages

The use of a suppressor typically increases signal-to-noise ratios about one order of magnitude. In addition to the obvious advantage of lower detection limits, other benefits of a greater signal-to-noise ratio include the ability to dilute dirty sample more, thereby extending column life; and the wider analytical dynamic range. Other advantages include the ability to use more concentrated eluents, which provides a greater range of elution control and the ability to use larger sample volumes; faster equilibration time; elimination of interference from counterions; and elimination of system peaks.

## Species Detected by Conductivity

Conductivity detection is typically the method of choice for ionic species whether they are inorganic or organic. This includes all anions and cations of strong acids and bases, such as chloride, sulfate, trifluoroacetate, sodium and potassium. Ions of weaker acids are detected provided they are significantly dissociated following suppression. As analyte ionization (dissociation) increases, so does sensitivity. For anion exchange chromatography, sensitivity is good for anions with a pKa below 6. Anions with a pKa above 7 are not detected. Fortunately, all organic acids with either carboxylate, sulfonate, or phosphonate functional groups have a pKa below 4.75, so conductivity is often the preferred detection method for these species.

Nearly all organic cations are amines. Aliphatic amines have a pKa around 10 and are easily detected. Aromatic and heterocyclic amines have a pKa between 2 and 7, too low to be detected by suppressed conductivity following cation exchange separation. Although nonsuppressed (single column) detection can be used for these species, sensitivity is generally poor. These amines are best detected by UV absorbance or by amperometric techniques.

## Eluents for Conductivity Detection

When choosing an eluent, the constraints placed by both the separation and detection methods must be considered. To obtain optimum separation, the elution strength of the eluent, the separation efficiency, and the resolution of the analytes of interest are the major criteria. For conductivity detection, the criteria for a good eluent are the relative conductivity response of the analytes and the magnitude of the background.

Eluents can be wholly aqueous containing only water and a strong electrolyte. Or, if an organic solvent compatible column such as a Dionex OmniPac™ or MPIC column is used, typical reversed phase solvents such as methanol or acetonitrile can be used. Organic solvents are essential components of ion-pair eluents and provide important selectivity control during ion exchange separations. Since these solvents are nonconducting, they do not interfere with conductivity detection.

## Anion Exchange

When a suppressor is used, the ionic components of the eluent must be such that they are removed or converted to weakly conducting compounds by the suppressor. Sodium salts of weak acids are used because they are converted to the neutral free acid form in the suppressor. The higher the acid's pKa, the lower the background conductivity following suppression. Weak acids with a pKa above 6 can be used for isocratic separations.

Sodium hydroxide solutions make excellent eluents for anion exchange because hydroxide is neutralized in the suppressor to water (the free acid form of hydroxide). Another commonly used eluent is a carbonate/bicarbonate buffer. It is suppressed to carbonic acid (pKa = 6.2). Carbonate/bicarbonate buffers are easily prepared and are routinely used for isocratic separations of inorganic anions.

Other eluents which may be used in suppressed anion chromatography are the sodium salts of boric acid (pKa = 9.2) and p-cyanophenol (pKa = 8.0). Eluents containing boric acid are effective at separating very weakly retained anions such as monoprotic carboxylic acids. Borate also forms weak bonds with hydroxy-organic acids, producing changes in selectivity as compared to hydroxide. p-Cyanophenol is a powerful monovalent displacer, useful for eluting strongly retained hydrophobic monovalent anions such as fumaric acid, iodide, and thiocyanate.

## Cation Exchange

A millimolar concentration of a dilute acid such as hydrochloric acid is a good choice for cation exchange chromatography of the monovalent cations: alkali metals and monovalent amines. However, hydrogen ion is too weak for divalent cations such as alkaline earth ions and divalent amines. These more strongly retained cations are eluted using 2,3-diaminopropionic acid (DAP). The ionization constants of the acid are 1.3, 6.7, and 9.4. At pH 1.3 (47 mM HCl) one-half of this acid is in the fully protonated divalent form, and one-half is in the partially protonated monovalent form. The divalent form has much stronger displacing power, making this a good eluent component for the elution of divalent cations. The membrane suppressor converts it to its zwitterionic form, which has no conductivity.

## Ion-Pair

Eluents for ion-pair chromatography contain mixes of aqueous and organic solvent solutions with hydrophobic ion-pair reagents as additives. Commonly used reagents are quaternary ammonium salts for anion separations, and long-chain sulfonates for cation separation. For suppressed conductivity detection, these reagents are easily used provided the counterions are either hydroxide (anion ion-pair) or hydrogen ion (cation ion-pair). These are provided by Dionex as IonSep reagents. They are purified solutions of quaternary ammonium hydroxide solutions and sulfonic acid solutions.

## Further Reading

1. Weiss, Joachim; Handbook of Ion Chromatography, Dionex, Sunnyvale, CA, 1985 (Urtext in Deutsch: VCH, Weinheim)

2. Small, Hamish; Ion Chromatography, Plenum, New York & London, 1990

# APPENDIX B
# GLOSSARY OF ION CHROMATOGRAPHY TERMS

**Analytical Column**
Synonymous with **Separator Column.**

**Band spreading**
The broadening of the sample band as it travels through the column. Band spreading can also occur in the injection valve, the detector cell, and interconnecting tubing.

**Calibration Curve**
A graph showing detector response in peak height or area versus analyte concentration.

**Capacity Factor (k′)**
The number of column volumes of eluent, pumped through the column, required to elute an analyte. Capacity factor is a dimensionless measure of retention independent of column length or eluent flow rate. It can be calculated as follows:

$$k' = \frac{t_r - t_o}{t_o}$$

Where $t_r$ = retention time
$t_o$ = retention time of unretained solute (column void volume)

**Cell Constant (k)**
A factor determined experimentally by measuring the conductance (G) of a standard solution of known equivalent conductivity ($\kappa$).

$$k = \kappa/G$$

The value of k depends upon the surface area of, and distance between, the electrode faces in the conductivity detector cell.

$$k = l/A$$

Where $l$ equals length, and A equals area of one electrode (the other electrode is equal to the first).

**Channeling**
The preferential flow of liquid along more open, less resistant paths through the column packing. This causes **Band spreading.**

**Column Efficiency (N)**
A measure of the *narrowness* of analyte bands as they elute from the column. High efficiency is desirable because resolution between closely spaced bands improves with greater efficiency. For a symmetrical (Gaussian) peak, column efficiency can be determined by the following:

$$N = 5.54(t_1/W_{1/2})^2$$

Where: $t_1$ = the peak retention time, in seconds

$W_{1/2}$ = the peak width at 1/2 height, in seconds

Column efficiency is proportional to column length: for a given resin and column diameter, increasing the column length increases the column efficiency. Synonymous with Theoretical Plates.

**Column Selectivity ($\alpha$)**
Describes the relative separation of the band maxima between two adjacent peaks. Selectivity can be determined by the following:

$$\alpha = (t_2 - t_0)/(t_1 - t_0)$$

Where: $t_1$ and $t_2$ = retention time of components 1 and 2, respectively.

$t_0$ = retention time of unretained components (void volume)

**Concentrator Column**
A short column used to retain and concentrate analytes from a measured volume of relatively clean sample. This allows large volumes of sample to be injected, lowering concentration detection limits.

**Conductivity**
A measure of the ease with which electrical current flows through a liquid contained between two oppositely charged electrodes. Conductivity is a characteristic of ions in solution. Units are Siemens.

**Counter Ion**
Ions carrying a charge opposite that of the sample ions (e.g., $Na^+$ may be the counter ion of a $Cl^-$ analyte. These ions preserve electrical neutrality in solution.

**% Crosslink**
Divinylbenzene content in a polystyrene/divinylbenzene (PS-DVB) resin; contributes to mechanical strength of resin and determines chromatographic properties.

**Equivalent Conductivity ($\lambda$)**
The contribution of an ionic species to the total conductivity of a solution measured in a standard cell having electrodes 1 $cm^2$ in area and exactly 1 cm apart.

**Guard Column**
Synonymous with **Pre-Column**.

**HETP (H)**
**H**eight **E**quivalent to a **T**heoretical **P**late. A measure of column efficiency which allows comparison between columns of different lengths.

$$HETP = H = L/N$$

Where: L = the column length (mm)

N = the number of theoretical plates

**Ion Exchange Capacity**
The number of active ion-exchange sites in a given weight or volume of resin. Most often expressed in meq/g or meq/mL.

**Ion Exchange Resin**
An insoluble polymer matrix containing fixed charged exchange sites (anionic or cationic). The resins used in IC are formed into small spherical particles (beads).

**Mho**
Obsolete unit of conductance, equal to $\Omega^{-1}$. Mho is replaced by siemens (S).

**Packing**
The material which fills a chromatographic column; usually a resin or silica-based material.

**Pellicular Resin**
A resin with a solid, non-porous core coated with a thin layer of more porous material. The exchange sites of pellicular ion exchange resins are located only on the surface layer of the bead. These resins have a low ion exchange capacity.

**Pre-Column**
A small column which prevents poisoning of the separator column by sorbing organic contaminants and removing particulates. It is filled with the same packing as the separator column. Synonymous with **Guard Column**.

**Regenerant**
A dilute acid or base which converts ion exchange sites in the membrane suppressor back to the form which will suppress the eluent conductivity.

**Resin**
An insoluble carbon-based polymer used as the structural foundation for chromatographic packings.

**Resolution (R)**
A measure of the separation between two sample components. It is expressed as the ratio of the distance between the two peak maxima to the mean value of the peak width at the baseline.

$$R = 2(t_2 - t_1)/(W_2 + W_1)$$

Where: $t_1$ and $t_2$ = the retention times of components 1 and 2, respectively

$W_1$ and $W_1$ = the baseline width of peaks 1 and 2, respectively (measured in the same units as the retention time).

R is proportional to the square root of efficiency (N).

A value of R = 1.5 represents "baseline separation" of the two peaks.

**Retention Time**
The time from injection to peak maximum; the basis for identification of a species in chromatography on a given system.

**Separator Column**
The column used to perform the actual chromatographic separations. It is different than guard column or precolumn; also called analytical column

**Siemen (S)**
Unit measure of conductance; the reciprocal of the electrical resistance of a solution.

**Suppressor**
A device used to minimize eluent conductivity and convert sample species to a common form, thus increasing detection sensitivity.

**Temperature Coefficient**
The percent of change in the conductivity of a solution with a $1^{\circ}$ C change in temperature. Every solution has a characteristic temperature coefficient which is determined experimentally.

**Theoretical Plates (N)**
See **Column Efficiency**.

**Void Volume ($V_0$)**
The volume occupied by the mobile phase in a packed column (this volume also includes the volume between the injection valve and the column and between the column and the detector cell). Unretained components are eluted in the void volume.

# APPENDIX C
# 4400 INTEGRATOR PROGRAMMING

## Introduction

The 4400 Integrator is a powerful tool for collecting and integrating data. With the addition of a DX-100 Automation Cable (P/N 43051 or 43069 - see Section 3.4.6.2) the integrator can also control and monitor the status of the DX-100 and Autosampler.

The Dionex 4400 Integrator Manual (Doc. 034212) contains installation and operation instructions for the integrator. Refer to Section 1, *Getting Started* in the integrator manual for instructions for initial installation and setup of the integrator. Once the integrator is installed, use the instructions in this Appendix and Section 3.4.6 of this manual to configure the 4400 to operate the DX-100 and, if installed, the autosampler. Section 3.4.6 provides instructions on connecting the signal wires and automation cable from the DX-100 to the integrator, and if applicable to the autosampler.

## Integrator Power-up Configuration

The power to the 4400 Integrator should be left on once it is set up. When the power is turned on, the Integrator performs self-diagnostic tests; the specific tests vary according to the options purchased with the Integrator. Refer to the 440 manual, Section 1, pages 1 through 7 for further explanation.

After the diagnostic test are run, a few parameters need to be defined to allow data to be collected correctly for the DX-100.

Press the *LCD STATUS* key; note that several parameters are displayed on the LCD:

**FI=1** indicates that you are using File 1.
**CS=1** indicates that the chart speed is 1 cm/min.
**ATTEN=1** indicates that the recorder response is 1 mV full scale.

Press the *PRINT FILE* key. Several parameters of the file are printed. **PW** stands for peak width; **PT** stands for peak threshold.

Set the following parameters to enable the 4400 Integrator to operate with the DX-100.

1. Press the CHART SPEED key and enter *0.5* (=0.5 cm/min).
2. Press the ATTEN key and enter *1024* (=1024 mV full scale).
3. Type in and enter *PT=1000* (a representative peak threshold for 1V signal).

## Setting Offsets

The signal offset from the DX-100 is set at the DIP switch panel (Section 4.1.2). The recorder offset for the integrator is given by the OF value. Dionex recommends that the factory default setting of 10% full scale be used for the DX-100, and that OF=0 be entered in the 4400 Integrator.

## System Testing

After the offsets have been entered via the DIP switches, the integrator is ready to operate in the area percent mode. This is a simple report format which records and integrates peaks from the DX-100 and then reports retention time and peak area. This mode is implemented by pressing *INJECT/END A* as the sample is being injected, and *INJECT/END A* to end the run and print the report.

If you are installing the DX-100 only, or are installing a new application, turn to Section 4, Operation, in this manual. When the application is installed and running, the retention time data can be used with Dialog in the Integrator to build a Method.

## Using DIALOG to Build a Method and Enter Time Functions

Once the application is running on the DX-100, an Integrator Method can be built to allow peak identification, calibration, and reporting. Time Functions can also be built into the Method to provide control of the DX-100 and autosampler.

Up to six Time Functions (T3 through T8) are available in the 4400 Integrator to control instrumentation. As described in Section 3.4.6 of this manual, the DX-100 Automation Cable is wired so that the five Time Function (TFN) commands that are used are assigned to the functions listed below. These TFN commands are programmed into the Method to occur at the appropriate times.

| Time Function (TFN) Command | Value | DX-100/ASM Function |
|---|---|---|
| T3 | 0 | ASM PROCEED |
| T3 | 1 | ASM START LOAD |
| T4 | 0 | DETECTOR RANGE NORMAL |
| T4 | 1 | DETECTOR RANGE × 10 |
| T5 | 0 | AUTO OFFSET OFF |
| T5 | 1 | AUTO OFFSET ON |
| T6 | 0 | SAMPLE LOAD |
| T6 | 1 | SAMPLE INJECT |
| T7 | 0 | PUMP OFF |
| T7 | 1 | PUMP ON |

After the integrator is powered up, and before the DX-100 *Automation* button is switched to the *Relay* position, the initial values of the Time Functions must be set. If they are not, it is likely that the pump will stop when the *Automation* button is switched to the *Relay* position. Initial values are set with the integrator TFN key. The default power- up values for the Time Functions are 0. This is appropriate for functions T3 through T6. T7 controls the pump and should be set to a value of 1 so the pump will continue to run when the system is switched to Relay Control. To enter the value for T7 press the TFN key and enter T7=1

The Method number chosen in Dialog determines the extent of peak identification, calibration, and reporting that the integrator performs. For most IC applications, it is appropriate to perform a multilevel calibration for each analyte. Calibration should also be performed with multiple injections at each calibration level.

Dialog Entry Example A, shown in Figure C-1, illustrates the setup of a Method for the analysis of five anions. The run is 10 minutes long, and the analytes are calibrated at three different concentrations, with two replicate injections at each level.

```
FI= 1.      FE= 1.      MN= 0.
PRESS 'ENTER' TO SKIP ENTRY
ENABLE BASELINE DRAWING? [Y/N] (N)
STORAGE MENU? [Y,N] (N)

FUNCTION NUMBER [0-10] (0)

FILE NAME () =" ANIONS
TIME        FUNCTION  VALUE        ①
TT= 0.01    TF=" T3   TV= 1
TT= 0.02    TF=" T3   TV= 0
TT= 1.40    TF=" T5   TV= 0
TT= 1.41    TF=" T5   TV= 1
TT= 1.50    TF=" T6   TV= 1
TT= 11.50   TF=" T6   TV= 0
TT= 11.50   TF=" ER   TV= 1
TT= 12.00   TF=" SR   TV= 1
TT=

METHOD NUMBER [0,1,2,5] (0) MN= 5
IF NV=0 THEN NO CALIB
IF NV=1 THEN NORMAL CALIB
IF NV>1 THEN MULTI-LEVEL CALIB
NUMBER OF LEVELS [0-26] ( 0. ) NV= 3
INJECTIONS/LEVEL (1) = 2
TOTAL CALIB INJECTIONS, RC= 6.
LINEAR[1] OR NONLINEAR[2] ( ) LS= 1
COMPONENT TABLE...
RET TIME        NAME
RT= 1.30        CN=" F
RT= 1.70        CN=" Cl
RT= 2.00        CN=" NO2
RT= 2.50        CN=" Br
RT= 3.40        CN=" NO3
RT= 7.90        CN=" SO4
RT= 3.80        CN=" HPO4
RT=
EXPECTED CONC [Y,N] (N)

RRT REF PEAK: RP(1)= 1.7
RRT REF PEAK: RP(1)=
MULTILEVEL CALIB VALUES:      ②
RETENTION TIMES:
  RT( 1)=    1.3       ( 2)=    1.7       ( 3)=    2.
CONCENTRATIONS:
  VA( 1)= 1      VA( 2)= 2      VA( 3)= 2
  VB( 1)= 2.5    VB( 2)= 5      VB( 3)= 5
  VC( 1)= 5      VC( 2)= 10     VC( 3)= 10
RETENTION TIMES:
  RT( 4)=    2.5       ( 5)=    3.4       ( 6)=    3.8
CONCENTRATIONS:
  VA( 4)= 2      VA( 5)= 2      VA( 6)= 2
  VB( 4)= 5      VB( 5)= 5      VB( 6)= 5
  VC( 4)= 10     VC( 5)= 10     VC( 6)= 10
RETENTION TIMES:
  RT( 7)=    7.9
CONCENTRATIONS:
  VA( 7)= 2
  VB( 7)= 5
  VC( 7)= 10
SAMPLE TABLE...
ANALYST () AN=" JOE
INJECTIONS/SAMPLE [0-254] ( 0. ) RA=
SAMPLES BETWEEN CALIB [0-254] ( 0. ) CI=
CONC UNITS () CU=" PPM
SAM IX   NAME          SAM AMT      SCALE
SI=
END OF DIALOG
```

**Figure C-1: Dialog Entry Example A**

①

**T3** is turned on and then off to allow the ASM to start loading and then to proceed with its normal cycling.
**T5** is turned off and then on to reset the AUTO OFFSET
T6 is turned on to INJECT the sample. It is turned off 10 minutes later, at the end of the run, to set the valve to LOAD.
**ER** is the command to end the run and generate a report.
**SR** is the command to automatically start the next run. 30 seconds is allotted between ER and SR to allow the report to print.

②

Concentrations are entered for the three calibration levels for each analyte. 1, 2.5, and 5 are entered for F. 2, 5, and 10 are entered for the other six.

To delete a Time Function while in Dialog, simply re-enter the TFN time, function, and value, but use a negative sign in front of the time. To review the Method, press the *PRINT FILE* key.

Once Dialog editing is finished, the integrator is ready for the first calibration run. If you are not prepared to start a calibration injection, type *CA=0* and press *Enter*. Additional runs may then be made before the calibration procedure is started. When you are ready to start calibration, type *CA=1* and press *Enter*.

Using the Time Functions in Example A, the integrator will repeat injections until the *ABORT A* key is pressed. This provides limited automation with no status monitoring of the DX-100 or autosampler. For a fully automated option, refer to the next section: BASIC Programming.

## BASIC Programming

The program presented in Figure C-2 can be used in conjunction with a Method program in Dialog. The advantages gained from using the BASIC program include monitoring of the DX-100 and ASM to see if they are ready to operate and to determine if a pressure alarm has been activated. The BASIC program also provides prompts to assist you in setting up the system to run a set of samples.

The program is typed in from the keyboard and, once entered, is started by typing and entering the word *RUN*. The 4400 Integrator Manual contains additional information on BASIC programming. The REM (Remark) statements in the program give some explanation of the statements used. Once the program is entered, it can be printed by typing and entering the word *LIST*. To edit a line, simply retype the line and press Enter.. To delete a line, simply type the line number and press *Enter*. The program is erased from memory if the 4400 Integrator power is turned off.

```
 10 REM  DX-100 AUTOMATION PROGRAM #1
  0 REM  VERSION 2.00   10 SEPT 91
 30 REM  PROMPTING FOR SYSTEM SETUP
 32 !!"CONFIRM THE AUTOMATION CABLE IS CONNECTED TO THE INTEGRATOR,"
 34 !"THE DX-100, AND THE AUTOSAMPLER."
 36 REM  "Aa" IS A NONSENSE VARIABLE TO ALLOW THE PROGRAM TO PAUSE
 38 INPUT "PRESS ''ENTER'' WHEN READY.",A
 40 !!"PLACE THE DX-100 INTO ''LOCAL CONTROL'' ."
 42 !"START THE PUMP AND ALLOW THE SYSTEM TO EQUILIBRATE."
 44 INPUT "PRESS ''ENTER'' WHEN EQUILIBRATED.",Aa
 50 REM  INITIALIZING VARIABLES
 52 St=0  REM  St = TOTAL # OF SAMPLES TO BE RUN
 54 Sr=0  REM  Sr = # OF SAMPLES WHICH HAVE RUN
 60 TFN"T3",0  REM  AUTOSAMPLER PROCEED/START LOAD
 62 TFN"T4",0  REM  RANGE NORMAL/*10
 64 TFN"T5",0  REM  AUTO OFFSET  OFF/ON
 66 TFN"T6",0  REM  INJECTION VALVE LOAD/INJECT
 68 TFN"T7",1  REM  PUMP OFF/ON
 70 TFN"T8",0  REM  UNUSED TFN
 80 !!"LOAD SAMPLES AND STANDARDS INTO THE AUTOSAMPLER AND SET TO ''RUN'' ."
 82 INPUT "PRESS ''ENTER'' WHEN READY.",Aa
 90 !INPUT "WHAT IS THE TOTAL NUMBER OF INJECTIONS TO BE MADE ?",St
 92 IF St>0 THEN 100
 94 !"NO TOTAL WAS ENTERED. TO TERMINATE PROGRAM, PRESS ''ESC'' ."
 96 GOTO 90
100 !!"PLACE THE DX-100 INTO ''RELAY CONTROL'' ."
102 INPUT "PRESS ''ENTER'' WHEN READY.",Aa
110 !!"THE SYSTEM IS READY TO BEGIN."
112 INPUT "TO START THE FIRST INJECTION PRESS ''ENTER'' .",Aa
120 REM  # OF SAMPLES RUN COMPARED TO TOTAL # OF SAMPLES
122 IF Sr=St THEN 212  REM  DIALOG TFN"GO" COMMAND MUST COME TO THIS LINE
130 REM  PRESSURE ALARM IS CHECKED
132 TFN"XD": XD(1)=PEEK#4C1F8
134 IF XD(1)=4 THEN 202
140 REM  AUTOSAMPLER STATUS IS CHECKED
142 TFN"XD": XD(1)=PEEK#1C1F8
144 IF XD(1)=0 THEN 142
150 REM  AUTOSAMPLER LOAD COMMAND GIVEN
152 TFN"T3",1
154 TFN"T3",0
160 REM  INTEGRATOR WAITS FOR "SAMPLE READY" FROM AUTOSAMPLER
162 TFN"XD": XD(1)=PEEK#3C1F8
164 IF XD(1)<3 THEN 162
166 IF XD(1)=3 THEN 172
170 REM  SAMPLE # INCREMENTED AND INJECTION COMMAND GIVEN
172 Sr=Sr+1
174 !!"INJECTING SAMPLE";Sr
175 !PT
176 INJECT A
200 REM  PRESSURE ALARM STATEMENT
202 !"PRESSURE ALARM DETECTED AFTER INJECTION OF SAMPLE";Sr
204 !"LOCATE THE CAUSE, CORRECT, AND TYPE ''RUN'' TO RESTART THE PROGRAM."
206 GOTO 220
210 REM  NOTICE OF RUN OR SCHEDULE FINISH
212 !"SCHEDULE FINISHED. TYPE ''RUN'' TO RESTART."
214 TFN"T7",0  REM  PUMP OFF
220 END
```

**Figure C-2: 4400 Integrator BASIC Program for the DX-100**

An example Method from Dialog is shown in Example B which illustrates a 10-minute run. Notice that no commands are given to the ASM in Dialog. These commands are performed by the BASIC program. Also notice that after the ER command a GO command is given. This replaces the SR command shown in Example A, and tells the 4400 Integrator to go to line 122 (you must use this line number) to start the next run.

```
FI= 1.      FE= 1.      MN= 0.
PRESS 'ENTER' TO SKIP ENTRY
ENABLE BASELINE DRAWING? [Y/N] (N)
STORAGE MENU? [Y,N] (N)

FUNCTION NUMBER [0-10] (0)

FILE NAME () =" ANIONS WITH BASIC
TIME        FUNCTION   VALUE
TT= 0.01    TF=" T5    TV= 1
TT= 0.02    TF=" T5    TV= 0
TT= 0.10    TF=" T6    TV= 1
TT= 10.10   TF=" T6    TV= 0
TT= 10.10   TF=" ER    TV= 1
TT= 10.60   TF=" GO    TV= 122
TT=

METHOD NUMBER [0,1,2,5] (0) MN=
```

**Figure C-3: Dialog Entry Example B**

# APPENDIX D
# SYSTEM SPECIFICATIONS

## • Analytical Pump

**Type:** Single reciprocating piston with metal-free flow path using PEEK components.

**Operation Mode:** Constant Volume.

**Flow Range:** 0.5 to 4.0 mL/min.

**Maximum Pressure:** 4000 psi (Pump Only).

**Temperature:** 40° C maximum.

## • Pulse Damper

**Type:** Mechanical high-pressure pulse damper with PEEK-based liquid conduit.
Maximum Pressure: 2400 psi.

## • Sample Injection

Pneumatically actuated three-way slider valve with interchangeable loops (25 mL provided) constructed of PEEK. 2400 psi nominal operating pressure. 4000 psi maximum operating pressure with increased pressure bolt torque. (See Section 5.3.4).

## • Pressure Alarms

**High Pressure:** User selectable via *Set High* switch from 1000 to 3800 psi in increments of 400 psi.

**Low Pressure:** User selectable via front panel *Low Limit On/Off* switch.

## • Conductivity Detector

**Type:** Analog signal processing.

**Cell Drive:** Pulsed bipolar fixed frequency.

**Full-Scale Output Ranges:** Eleven separate ranges, 0.01 µS to 1000 µS, set via front panel button switches.

**Auto Offset:** 0.0 to 1000 µS.

**Filtering options for Nonsuppressed IC:** Three user selectable filtering settings for optional use with non-chemically suppressed applications.

**Temperature Compensation:** Selectable compensation between 1.3% and 2.0% per 1 C°. Sensitive thermistor directly in the flow stream 2.5 mm from electrodes responds rapidly to temperature change.

**Analog Signal Output Voltage:** User selectable full-scale recorder output of 10, 100, or 1000 mV.

**Control Modes:** Local or remote via relay closures.

## • Detector Flow Cell

**Electrodes:** MicroMass passivated 316 stainless steel electrodes for accurate temperature compensation.

**Cell Body:** Chemically inert polymeric material.

**Active Volume:** 1.25 µL.

**Maximum Operating Pressure:** 300 psi (20 atm)

## • Modes of Operation

**Manual:** User sets parameters via front panel and makes an injection using the front panel injection button.

**Automated:** Integrator control of the DX-100 inputs, or advanced integrator control and monitoring of both the DX-100 and Dionex Autosampler.

## • Column Configurations

**One Column Set:** Standard mounting configuration is for one set of columns (analytical and guard), a MicroMembrane Suppressor, and an optional thermal stabilizer (TS-1) to be mounted on the inside of the front door. Columns and suppressors are not included with the system.

**Two Column Set:** Optional configuration has two sets of columns and suppressors. The two column option includes valving to easily change from one application to another.

## • Front Panel Display and Controls

**High/Low pressure alarm**
**Bar graph display of current operating pressure in MPa or psi.**
**Display and Setting of full scale conductivity range.**
**Numeric display of conductivity in µS.**
**Momentary numeric display of mS Offset, MPa, psi.**
**Chart recorder control.**
**Relay/Local control**
**Pump On/Off**
**Auto Offset On/Off**
**Sample Inject/Load**

## • Physical

**Power Requirements:** Available in 100V/50 Hz, 115V/60 Hz, 220V/50 Hz, 240V/50 Hz.

**Operating Power:** 75 VA

**Maximum Current with Accessory Loads:** 10 A

**Operating Temperature Range:** 10° to 40° C (50° to 104° F).

**Dimensions:** (H × W × D): 72 cm × 36 cm × 48 cm (28.3" × 14.2" × 18.9").

**Weight:** 33.6 Kg (74 lb).

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